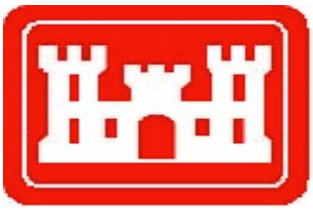
FINAL
REMEDIAL INVESTIGATION QUALITY ASSURANCE PROJECT PLAN

For

FORMERLY USED DEFENSE SITE
CHARLESTON AIR FORCE STATION
CHARLESTON, MAINE
D01ME011201

Contract No.: W912WJ-19-D-0002
TASK ORDER F0182

Prepared for:



New England District
U.S. Army Corps of Engineers
696 Virginia Road
Concord MA 01742-2751

29 September 2020

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This is to certify that Wood has performed a peer technical review of this deliverable under USACE NAE Contract No. W912WJ-19-D-0002 *consistent with Wood Quality Management Program Procedure-PJM-PRO-002, Technical Review.*

Peter S. Baker, CG
Project Manager

Scott F. Calkin, PG
RI Technical Lead

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ACRONYMS AND ABBREVIATIONS

AFS	Air Force Station
cis-1,2-DCE	cis-1,2-Dichloroethene
DERP	Defense Environmental Restoration Program
DoD	Department of Defense
DP	Drive Points
DQO	Data quality objective
EPH	Extractable Petroleum Hydrocarbons
FS	Feasibility Study
ft	foot/feet
FUDS	Formerly Used Defense Site
FUDSChem	Formerly Used Defense Site Chemical Database
FUDSMIS	Formerly Used Defense Site Management Information System
INPR	Inventory Project Report
ISM	Incremental Sampling Methodology
MEDEP	Maine Department of Environmental Protection
µg/L	Micrograms Per Liter
NAE	New England District
NCO	Non-Commissioned Officer
NE	Northeast
NW	Northwest
PA	Preliminary Assessment
PCB	Polychlorinated Biphenyl
PDT	Project Delivery Team
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SE	Southeast
SS	Surface Soils
SVOC	Semivolatile Organic Compound
SW	Southwest
TCE	Trichloroethene

UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
USAF	United States Air Force
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VOC	Volatile Organic Compound
VPH	Volatile Petroleum Hydrocarbons
Wood	Wood Environment & Infrastructure Solutions, Inc.
XRF	X-ray fluorescence

1.0 Introduction

Wood Environment & Infrastructure Solutions, Inc. (Wood) has prepared this Remedial Investigation (RI) Quality Assurance Project Plan (QAPP) in support of the RI of the former Charleston Air Force Station (AFS or Site) located in Charleston, Maine. The format of this document is based on the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP, March 2005) and the Optimized UFP-QAPP Worksheets (March 2012). This QAPP and the associated RI Sampling and Analysis Plan together define investigation objectives, procedures, and quality assurance and quality control (QA/QC) for this program. This project will be completed in accordance with the Performance Work Statement, Remedial Investigation/Feasibility Study (RI/FS) prepared by the United States Army Corps of Engineers, New England District (USACE-NAE) dated April 2019 and any subsequent modifications and additional scope changes developed by USACE-NAE. USACE engineering objectives identified in USACE engineering manuals EM 1110-1-4007, EM 385-1-1, ER 1110-1-263, EM 200-1-6, and EM 200-1-10 are applicable to activities completed under this QAPP. This QAPP has been prepared for the USACE-NAE under contract number W912WJ-19-D-0002 Task Order F0182 and defines investigation objectives, procedures, and QA/QC for this program. RI work will be conducted under the Department of Defense (DoD) Defense Environmental Restoration Program (DERP) Formerly Used Defense Site (FUDS) program. Under the FUDS program Charleston AFS is designated as Property Number D01ME0112.

The following sections provide an overview of the Site description, setting and history. A more detailed description of these elements is provided in the RI Sampling and Analysis Plan (SAP).

1.1 Site Description

The former Charleston AFS consisted of 82.3 acres in Charleston, Maine, located in Penobscot County. The property is on State Highway 15, approximately 25 miles northwest of Bangor, ME and 2.5 miles east of Charleston, ME.

The Air Force built Charleston AFS as an Aircraft Control and Warning Station and became fully operational on 01 June 1952. Its radars scanned the offshore airspace of the Bangor Defense Area, searching for incoming enemy aircraft. In May 1959, the station converted to a semi-automatic ground environment system, and it began backup interceptor operations in March 1963. Exhibit 1 is a historic aerial photograph of the facility. The Air Force declared Charleston AFS surplus on 29 September 1979.

Charleston AFS is currently owned by the State of Maine and is occupied by the Mountain View Youth Development Center and the Charleston Correctional Facility. The public has limited access to portions of the former Charleston AFS. The developed portions of the corrections facility have security personnel and fencing with secured entries. However, the area east of Highway 15 (former radio receiver and Non-Commissioned Officers [NCO] club) and the abandoned radar facilities can be entered on foot from adjacent properties. It is anticipated the former Charleston AFS will continue to be used by the Corrections Facility for the foreseeable future.

Exhibit 1 Presents a historical aerial photograph (unknown date) of the Site.

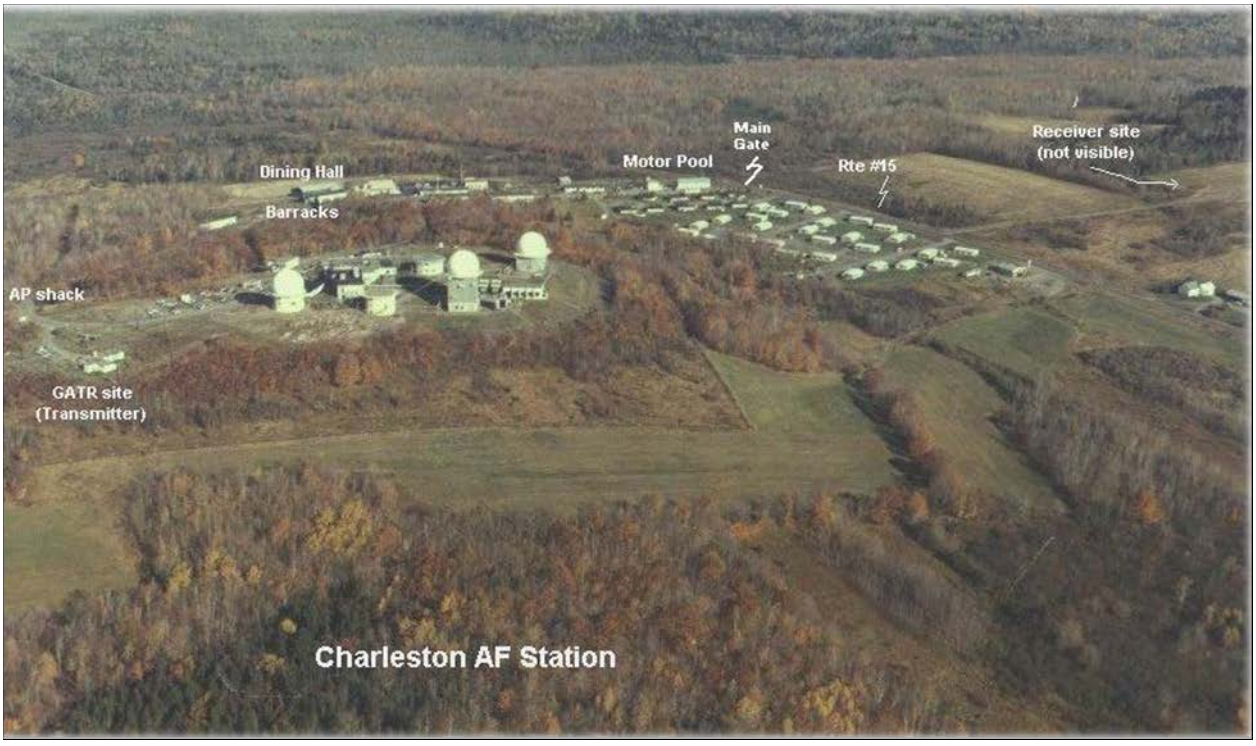


Exhibit 1 Charleston Air Force Station (date unknown)

1.2 Summary of Previous Work

There have been no DoD soil and groundwater investigations completed at the former Charleston AFS. A removal action of transformers and some Underground Storage Tanks (USTs) was completed.

The New England District of the U.S. Army Corps of Engineers prepared an Inventory Project Report (INPR) for the Charleston AFS (D01ME011200) on 07 November 1988. The report which was approved on 20 January 1989, identified one DERP-FUDS project on the property. The project consisted of removal of twenty-two transformers and twenty-seven 275-gallon underground heating fuel storage tanks, and three 10,000-gallon underground fuel storage tanks. Based on information on Formerly Used Defense Site Management Information System (FUDSMIS), this project was completed in 1990, and in addition to the tank and transformer removal, the contractor removed 18 tons of petroleum-contaminated soil, 25 cubic yards of Poly-Chlorinated Biphenyl (PCB)-contaminated soil, and 340 tons of waste oil-contaminated soil. The UST removal action was conducted by the Maine Department of Environmental Protection (MEDEP).

Preliminary Assessment Report

A Preliminary Assessment (PA) Report was prepared for the Site by the USACE in January 2018 (USACE, 2018). The summary of the PA findings are as follows:

- A small arms firing range was present.
- Three 10,000-gallon USTs were present on the Hilltop Area where radars were located. In 1987, MEDEP testing indicated the presence of PCBs, petroleum and waste oil in the tanks and surrounding soils. Note: additional research showed that one of these USTs was 8,500 gallons.
- In 1989 the two 10,000-gallon USTs, the 8,500 gallon UST and the twenty-one 275-gallon heating fuel USTs, two well house gasoline tasks and 22 transformers were removed and disposed by MEDEP.
- During the 1989 removal action approximately 18 tons of petroleum contaminated soil, 25 cubic yards of PCB contaminated soil and 340 tons of waste oil contaminated soil was removed. Little to no documentation of the removal action and no confirmation sampling data is available. No investigation of groundwater was undertaken or reported.
- An UST is suspected to be present at the radio transmitter building/NCO club on the adjacent property to the east.
- A bedrock water supply well drilled by the State of Maine and groundwater sampled was found to contain trichloroethene (TCE) at a concentration of 3.4 Micrograms Per Liter (µg/L) and cis-1,2-dichloroethene (cis-1,2-DCE) at a concentration of 3.5 µg/L, which are just below the Maximum Contaminant Level and Maine Exposure Guideline of 5 µg/L and 10 µg/L, respectively. This well is located outside the former property boundary of the Charleston AFS and currently owned by the Correctional Facility.
- The PA identified several other possible source areas in the former industrial support area.

Technical Memorandum Historic and Aerial Photography Analysis/Research

During the planning phase of this RI, Wood identified additional potential source areas following a review of historical air photography, existing site drawings and microfiche as-built drawings. This information and a summary of the findings are documented in Final Technical Memorandum Historic and Aerial Photography Research/Analysis (Wood, 2020a). The Technical Memorandum is provided in RI SAP. These areas include:

- Former coal storage pile runoff pond
- Dry well associated with a wash rack
- Former Fire Station
- Discharge drain associated with the former Auto Storage Building
- UST (2,000-gallon fuel oil) associated with the Motor Pool
- 2,000 gallon tank in the industrial area
- Septic systems serving the former Operations Building, Building 211, Building 212, and Building 213
- 275-gallon waste oil storage tank near the former Operations Building
- Discharge lines serving Buildings 212 and 213

- Two 40,000-gallon diesel fuel oil USTs serving Building 213; abandoned in place
- Lube oil tank serving Building 213
- Waste oil and possibly PCBs observed in the excavation during a water line repair on the Hilltop, 200 feet (ft) downslope of the former three USTs (two 10,000 gallon and one 8,500 gallon) on the Hilltop. This event triggered the investigation, sampling, and removal of the USTs.
- Transformer locations in the industrial area

QAPP Worksheet #1 & 2: Title and Approval Page

(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

Site Name/Project Name: Charleston Air Force Station/Remedial Investigation

Site Location/Number: Charleston, Maine/Project No. D01ME011201

Contract/Work Assignment Number: W912WJ-19-D-0002

Lead Organization: United States Army Corps of Engineers, New England District

Project Manager: Harry R. Hendler
harry.r.hendler@usace.army.mil

Signature

Lead Investigation Organization: Wood Environment and Infrastructure Solutions

Project Manager: Peter S. Baker
peter.baker@woodplc.com

Signature

Quality Assurance Manager: Julie A. Ricardi
julie.ricardi@woodplc.com

Signature

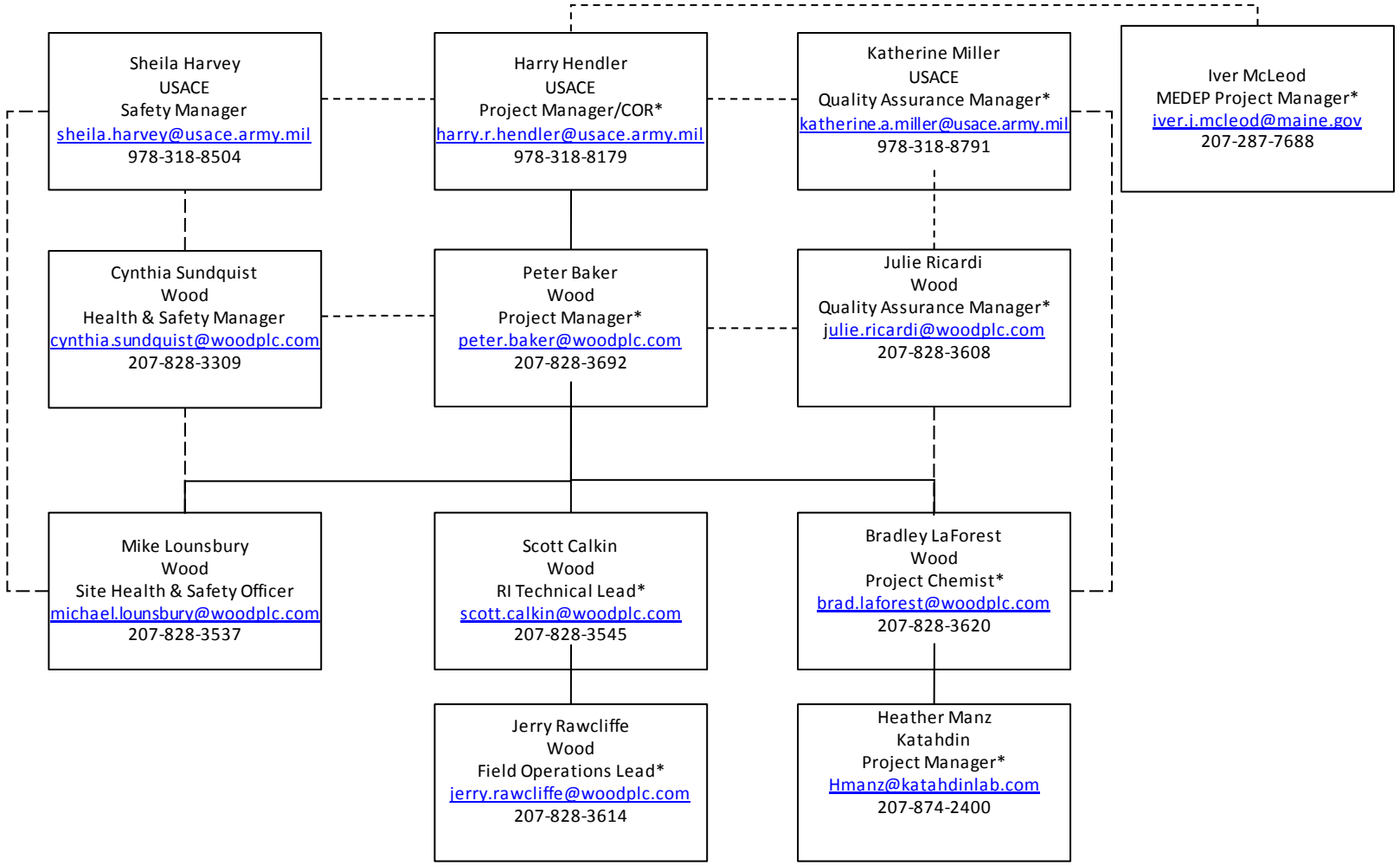
State Regulatory Agency: Maine Department of Environmental Protection
Project Manager: Iver J. McLeod
iver.j.mcleod@maine.gov

Signature

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution

(UFP-QAPP Manual Section 2.3 and 2.4)(EPA 2106-G-05 Section 2.2.3 and 2.2.4)

*QAPP recipient Lines of authority _____ Lines of Communication ----------



QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet

(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

ORGANIZATION: Wood

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Jeff Pickett	Program Manager	B.S. Geology/38 years	CG	
Peter Baker	Project Manager	B.S. Geology/38 years	CG	
Scott Calkin	RI Technical Lead	M.S. Geophysics and Geology/32 years	PG	
Julie Ricardi	Quality Assurance Manager	B.S. Chemical Engineering/ 35 years		
Bradley LaForest	Project Chemist	B.A. Biology/29 years	CEAC	
Jerry Rawcliffe	Field Operations Lead	B.S. Geologic Sciences/36 years		

ORGANIZATION: Katahdin Analytical Services

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Leslie Dimond	Quality Assurance Manager	B.A. Chemistry/26 years	NA	
Heather Manz	Project Manager	B.S. Earth Sciences/10 Years	NA	

*Signatures indicate personnel have read and agree to implement this QAPP as written

Notes:

B.A. = Bachelor of Arts
B.S. = Bachelor of Science
CG = Certified Geologist
CEAC = Certified Environmental Analytical Chemist
M.S. = Masters of Science
PG = Professional Geologist

QAPP Worksheet #6: Communication Pathways

(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory agency interface	USACE	Harry Hendler	harry.r.hendler@usace.army.mil 978-318-8179	Is the primary point of contact for regulatory interface. With USACE approval, contact may be made by the Wood Project Manager or Wood Program Lead through telephone or electronic mail and a record will be retained detailing the correspondence.
Field progress reports	Wood	Jerry Rawcliffe	jerry.rawcliffe@woodplc.com 207-828-3614	The Wood Field Operations Lead must notify the Wood RI Technical Lead, who in turn notifies the Wood Project Manager (verbal, electronic), who in turns notifies USACE (verbal, electronic).
Stop work due to safety issues	USACE, WOOD, Subcontractors	On-site personnel	jerry.rawcliffe@woodplc.com 207-828-3614	On-site personnel must notify the Wood Site Health and Safety Officer, who in turn notifies the Wood Program Lead and the Wood Project Manager (verbal, electronic), who in turns notifies USACE (verbal, electronic).
QAPP changes prior to field work	Wood	Julie Ricardi	julie.ricardi@woodplc.com 207-828-3608	The Wood Quality Assurance Manager notifies the Wood Project Manager, who in turn notifies USACE (verbal, electronic). Also notifies the Wood Program Lead.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
QAPP changes during project execution	Wood	Jerry Rawcliffe	jerry.rawcliffe@woodplc.com 207-828-3614	The Wood Field Operations Lead will notify the Wood Quality Assurance Manager who will notify Wood Project Manager who in turn notifies USACE.
Field corrective actions	Wood	Scott Calkin	scott.calkin@woodplc.com 207-828-3545	Field corrective actions will be developed by the Wood RI Technical Lead, the Wood Project Manager and the Wood Quality Assurance Manager. Corrective Actions will be communicated to the Wood RI Technical Lead, the Wood Quality Assurance Manager, and to the Wood Field Operations Lead, who will communicate corrective actions to the field team.
Sample receipt variances	Katahdin	Heather Manz	hmanz@katahdinlab.com 207-874-2400	The Katahdin Project Manager will notify the Wood Project Chemist within 24 hours of sample receipt, who will then notify the Wood Quality Assurance Manager and the Wood Project Manager. The Wood Quality Assurance Manager and the Wood Project Manager will implement field corrective action (if needed) within 24 hours of when the notification occurs.
Laboratory quality control variances	Katahdin	Heather Manz	hmanz@katahdinlab.com 207-874-2400	The Katahdin Project Manager will notify the Wood Project Chemist and the Wood Quality Assurance Manager within 24 hours of variance. If quality control variances contradict the minimum requirements of the DoD Quality Systems Manual, then the Wood

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
				Quality Assurance Manager and the Wood Project Manager will contact USACE to discuss and receive approval for the variances within 7 days of notice of variance.
Analytical corrective actions	Wood	Bradley LaForest	brad.laforest@woodplc.com 207-828-3620	The Wood Project Chemist will respond to issues from Katahdin with potential corrective action (verbal, written or electronic) within one week of notification.
Data verification issues, e.g., incomplete records	Wood	Bradley LaForest	brad.laforest@woodplc.com 207-828-3620	The Wood Project Chemist will resolve any data verification issues with Katahdin within one week of issue being identified.
Data validation issues, e.g., non-compliance with procedures	Wood	Bradley LaForest	brad.laforest@woodplc.com 207-828-3620	The Wood Project Chemist will resolve any data validation issues with Katahdin within one week of issue being identified.
Data review corrective actions	Wood	Bradley LaForest	brad.laforest@woodplc.com 207-828-3620	The Wood Project Chemist will communicate necessary data review corrective actions with Katahdin within one week of corrective actions.

QAPP Worksheet #9: Project Planning Session Summary

(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)

Date of planning session: December 10th, 2019

Location: 511 Congress Street, Portland, Maine

Purpose: Charleston AFS Scoping Meeting - USACE Provides Overview of RI Scope and Tasks Agreed Upon with the MEDEP. Wood Presents Current Understanding of Potential Source Areas

Participants:

Name	Organization	Title/Role	Email
Harry Hendler	USACE	Project Manager	Harry.r.hendler@usace.army.mil
Ken Heim	USACE	Hydrogeologist	Kenneth.j.heim@usace.army.mil
David Oster	USACE	Ecological Specialist	David.a.oster@usace.army.mil
Peter Baker	Wood	Project Manager	Peter.baker@woodplc.com
Scott Calkin	Wood	RI Technical Lead	Scott.calkin@woodplc.com
Bradley LaForest	Wood	Project Chemist	Brad.laforest@woodplc.com
Charles Lyman	Wood	Project Biologist	Charles.lyman@woodplc.com
Amy Quintin	Wood	Human Health Risk Assessor	Amy.quintin@woodplc.com
Tony Rodolakis	Wood	Ecological Risk Assessor	Tony.rodolakis@woodplc.com

Meeting Notes/Comments:

Harry Hendler kicked off meeting by providing an overview of the Monday meeting between COE and MEDEP. MEDEP is generally OK with the proposed investigation, but strongly requested sampling of private wells. Gail Lipfert (MEDEP) is reviewing historical information and may provide additional comments.

Peter Baker presented historical information that provided locations of areas of potential concern including motor pool, auto storage building, CE maintenance building, water treatment plants, septic tanks, septic sand filter, chlorinator building/outfall, boiler plant, coal pile, possible coal ash pond, septic tanks/leachfields at 3 buildings on hilltop, MEDEP photos from UST and soil removal

of three 10,000 gallon USTs showing excavation and stockpiling of soil on north side of access road.

USACE went through major changes to the original investigation that were discussed with MEDEP, including:

- Major change to groundwater program – USACE will agree to test private wells, but for limited analysis – 4 rounds. Would like us to "justify" which private wells we should sample. Estimate around 5 locations within approximately one half mile of the site. Wanted to know if there is a way to estimate flow directions to rule out possible receptors? Wood should address this question, if possible in the work plan.
- Testing of seeps (not in original proposal) – Will do 4 rounds of 4 samples in 4 quadrants (if seeps are present).

USACE presented the following recommended scope and there was a group discussion of each following areas.

Transformer Pad Samples:

- Surface soil (SS) samples, around each pad.
    - Pads 1-10 = 4 SS/pad
    - Composite SS vertically, hand augered = 0-12"
    - PCB only
- Concrete chip samples will be collected for PCBs on transformer pads only if showing visual evidence of contamination/residue. Need to identify what the data will be used for. Are there regulations for leaving PCB -contaminated building materials in place vs removal. There are no human health criteria that Wood is aware of for this (no standard exposure pathway). Wood will look into state regulations further.

Transformer Pole Samples:

- Composite SS, hand augered = 0-12"
- PCB only
- USACE noted that many poles are present on the hilltop and concerned that information on which poles had transformers is not available. We need to either justify sampling select poles (poles only selected if we have evidence of transformer positions) or consider incremental sampling methodology (ISM)/composite samples to cover all poles – one sample per pole. Noted that state has a preference for ISM.
- Only concerned about transformer poles on hilltop not transformer poles former industrial/housing area.

Filled/Abandoned in Place two 40,000 UST location:

- Geophysical survey to outline the perimeter – Wood indicated that we had done a ground penetrating radar survey during site visit and had located and staked the 2 USTs.

- Discussed USACEs recommendation for soil gas survey of area. Wood noted that these were diesel fuel tanks and it has been 40 years since they were used, so soil gas for Volatile Organic Compounds (VOCs) might not be the best method for detecting the presence /absence of residual fuel contamination. USACE noted that MEDEP wanted to have samples from the soils at the bottom of the tanks. Agreed that we need to do soil borings and sampling.
- Wood will use their expertise to confirm the analytical parameter list based on an assumed UST contents.

Former three 10,000 gallon UST location

- Geophysical survey
- 5 Drive Points (DP) confined to former UST area.
- 2 samples per DP – one at bottom of boring and one near mid-point
- Discussed MEDEP photo documentation that showed stock piling of soil on north side of road across from tank/soil excavation area during the tank and soil removal conducted by MEDEP. Wood recommended sampling of soils in this area. USACE does not want to do sampling in this area at this time. The fact that MEDEP did the removal and could be responsible for this issue complicates the approach. Will follow USACE direction on this item.
- If soils present an issue on the surface, then will refer this area as a PRP project to be addressed separately from this RI.

Abandoned Drums:

1 SS underneath each drum location for VOC, Semivolatile Organic Compounds (SVOCs), metals, Extractable Petroleum Hydrocarbons (EPH) and Volatile Petroleum Hydrocarbons (VPH).

- Composite SS = 0-12”

Additional 5 Drive Points:

- 1 DP per dome at the dome entrance – most likely area where disposal of waste may have occurred
- 1 soil sample at soil/bedrock interface for VOCs
- Add in geophysics, borings and sampling for three septic tanks/leachfields at Buildings 211, 212, 213

Small Arms Range:

- 48 field X-ray fluorescence (XRF) measurements within the footprint of the berm/target.
  - 10 measurements in the field between the firing line and the target/berm,
  - 20 measurements within the target/berm,
  - 10 measurements behind the target/berm, and

- 8 background measurements outside of the line of fire

Heating Facility/Steam Plant:

- 2 DP – one upgradient of boiler plant and one downgradient of fuel tanks

Former Auto Maintenance Shop/Motor Pool:

- 1 DP sample location in the grassy median to the south
- 1 DP sample location back of the former building where waste disposal may have occurred
- Sample Depth = TBD
- VOC, SVOCs, metals, EPH and VPH

Auto Storage Building:

- 1 DP sample near the hillside to the north outside of the asphalt parking lot.
- Sample Depth = TBD
- VOC, SVOCs, metals, EPH and VPH

WWTP Septic Sand Filter:

- 3 DP sample locations on the berm of the retention pond.
- Sample Depth = TBD
- VOC, SVOCs, metal, RAD, EPH, VPH

WWTP and Septic Tank

- 3 DP sample locations, 1 downgradient of septic tank, 1 downgradient of WWTP (possible coal ash pond area) , 1 southeast of former WWTP (Weld shop)
- VOC, SVOCs, metal, RAD, EPH, VPH

Former Radio Receiver Building

- Geophysical survey to locate possible USTs
- If a possible UST is identified, then sample around the perimeter of the UST
- AST/drum area – 4 DPs
- VOCs, EPH, VPH

Groundwater Investigation:

- Sample four existing on-site wells before treatment, if possible
    - Wood to request existing State well sampling results from Sam Bradeen (Mt View Correctional Facility Maintenance Supervisor)
    - Include existing wells 1, 2,3 and former restaurant well

  - discharge well development/purge water into storm water pond next the former restaurant if possible
  - 4 rounds/well
- Collect seep samples for VOCs
  - Approximately 4 quadrants (quads)
  - 4 samples per quad (quads measured in line around hilltop in 625 ft. segments. Tentative number of samples = 16 but adjust up or down based on number of seeps identified.
  - 4 rounds
- Sampling of private residential wells within approximately one half mile of the site – 4 rounds

Wood Risk Assessors - Amy Quintin and Tony Rodolakis will schedule a call with USACE Risk Assessors - Cindy Auld and David Oster before have full project delivery team (PDT) work plan scoping meeting (see below) and write up the risk assessment work plan to confirm the approach, and report structure for the risk assessment. Chemists should also be on the call. Need to come to a consensus on the laboratory analysis required for the area to be investigated. Note, this needs to happen to confirm the approach before the workplans are developed.

USACE indicated the process would be for Wood to draft and USACE to agree to the work plan, then we will modify contract to incorporate the proposed changes. Meeting proposed in January to confirm with COE PDT the scope of work and then Wood will prepare the Work Plan.

Schedule for Draft Work Plan – Shooting for Mid-Feb but might be tight. Work plan preparation will follow PDT scoping meeting in January. USACE will need to brief ITR before we can have meeting with PDT. This will require revised scope of work from Wood. Wood will prepare figures/table that outlines the revised scope of work and submit to USACE. If possible USACE would like to sample seeps in the spring. Asked if this portion of the work plan could be provided separately to expedite the work.

Action Items:

Action	**Responsible Party**	**Due Date**
Revise Performance Work Statement and Modify Contract	Harry Hendler	TBD
Revise Scope of RI Work Plan	Peter Baker, Scott Calkin, Brad LaForest	TBD
Review Microfiche Supplied by the USACE	Peter Baker and Scott Calkin	Complete before January Scoping Meeting

Action	Responsible Party	Due Date
Revise Risk Assessment Approach	Cindy Auld, David Oster, Amy Quintin, and Tony Rodolakis	Complete before January Scoping Meeting
Revise and Confirm Analytical Program	Brad LaForest and Kathy Miller	Completed before January Scoping Meeting

Date of planning session: January 22nd, 2020

Location: USACE – NAE, Concord, MA

Purpose: Charleston AFS Scoping Meeting – Independent Technical Review (ITR) Introduction to Project, Resolve RI Technical Approach, Risk Approach, and Analytical Program

Participants:

Name	Organization	Title/Role	Email
Harry Hendler	USACE	Project Manager	Harry.r.hendler@usace.army.mil
Ken Heim	USACE	Hydrogeologist	Kenneth.j.heim@usace.army.mil
David Oster	USACE	Ecological Specialist	David.a.oster@usace.army.mil
Cindy Auld	USACE	Human Health Risk Assessor	Cynthia.a.auld@usace.army.mil
Amy Rosenstein	USACE	ITR	Amy.b.rosenstein@usace.army.mil
Dabra Seiken	USACE	ITR	Dabra.i.seiken@usace.army.mil
Kathleen Atwood	USACE	ITR	Kathleen.a.atwood@usace.army.mil

Name	Organization	Title/Role	Email
Kathy Miller	USACE	Project Chemist	Katherine.a.miller@usace.army.mil
Joshua Mulvey	USACE	Real Estate	Joshua.I.Mulvey@usace.army.mil
Peter Baker	Wood	Project Manager	Peter.baker@woodplc.com
Scott Calkin	Wood	RI Technical Lead	Scott.calkin@woodplc.com
Bradley LaForest	Wood	Project Chemist	Brad.laforest@woodplc.com
Amy Quintin	Wood	Human Health Risk Assessor	Amy.quintin@woodplc.com
Tony Rodolakis	Wood	Ecological Risk Assessor	Tony.rodolakis@woodplc.com

Meeting Minutes

1. Introductions
2. Harry Hendler – Introductory remarks. Independent Technical Review (ITR) is present to understand genesis and rationale for project decisions and activities to expedite reviews. USACE has meet with regulators on approach of remedial investigation. Regulators are ok with the overall approach but await details.
3. Harry Hendler – Site overview and history for benefit of ITR. Driving force of RI is identifying the source and extent of the TCE and DCE detection in a single production well at the prison facility. This well is still in use and is diluted with the other three production wells. [remaining history discussion not fully summarized here]
4. Harry Hendler – Reviews meeting minutes from January 7th Conference Call.
    a. In FUDS programs, there are phases. We are in the RI/FS phase. Current contract is for RI. So activities and report will be called an RI, not an SI and not a Phase I RI. We will use the objectives within the report to clarify that the objective will be to initially determine the presence/absence of contaminants for most areas so as not to mislead readers/public/regulators. This round of sampling is primarily to evaluate which areas require more sampling to delineate nature and extent due to lack of data. The RI report should be a document that clearly identifies what was

accomplished, and what is left to accomplish regarding determination of nature and extent of contamination.

b. Wood will generate a SAP and QAPP. Titles will be RI SAP, RI QAPP. Data quality objective (DQOs) within the QAPP will clarify intent as presence/absence or characterization per area. Characterization may be feasible within certain areas such as the firing range and the USTs, whereas others are clearly presence/absence (transformer poles etc.)
c. Cindy Auld – The majority of sampling is likely to be biased during this round, and therefore inappropriate for generating an average or range of exposure. Therefore, the risk components in this "RI" will be limited to a comparison to screening levels. Site-specific background values for metals and PAHs will be useful in understanding and identifying relevant contaminants pursue in the next phase of sampling.
d. General Discussion from USACE - Pesticides will not be sampled. No indication that pesticides were stored or mixed at Charleston.
e. Peter Baker – Explanation of a "Stringent" site under the MEDEP petroleum regulatory requirements. MEDEP categorizes and regulates petroleum release sites differently based on distance between the release to private water supplies and other factors. The closer to a drinking water source, the more stringent the cleanup actions. Current Maine DEP guidance follows a tiered risk-based approach. During a UST removal action at the former motor pool, the motor pool was classified by MEDEP as a "Stringent" site because of proximity to facility production wells at facility. Stringent sites require remediation of soil and groundwater based on EPH/VPH concentrations.
f. Harry Hendler – Confirms that CERCLA needs to meet standards of other regulated programs, ARARS/TBCs need to include the Maine requirements. We are the prime, therefore we lead the decision. If waste oil contains CERCLA regulated chemicals such as benzene or PCBs tank cleanup can be pursued under CERCLA. If CERLCA chemicals are absent, but EPH/VPH are present, we will need to follow substantive Maine UST closure requirements. If there is actionable risk from EPH/VPH, it will require direction from USACE in-house council and Programs before engaging the State.

5. Harry Hendler – Reviewed Summary of Sampling and Analysis table from Wood with Peter Baker and Scott Calkin [full details of discussion captured in revised table to be provided by Harry– overview is provided below of major discussion points]
    a. Background data – PAHs and metals at 20 locations. TOC and pH will also be analyzed to eliminate organics and some metals as COPC in the ERA. Only metals and PAHs via SIM. No groundwater background.
    b. PCBs – Soil PCBs to be analyzed in two steps. First, analyze for Aroclors, then based on findings, identify a subset for congener analysis to give a more refined estimate of which PCBs are present (some drive risk more than others (e.g. dioxin-like PCBs) to inform future sampling. The number of congener analysis was revised to be a subset of the total number of PCB analysis. Concrete PCBs to be analyzed only for Aroclors. QAPP to note that soil congeners are needed for HHRA and ERA, and that concrete PCBs are needed only for disposal characterizations.

c. SVOCs – Discussion between USACE regarding whether to analyze only for PAHs or for the full 8270 SVOC suite. SIM is required as we need to meet low detection limits.
    i. Dabra Seiken – If not a FUDS release, we may not technically be funded to look for full SVOC suite. Corps PMs have been asked to reduce compound lists and not collect irrelevant data. Dabra indicated that she has not seem non PAHs compounds to be present at other sites, with the exception of phthalates, which are usually written off as common lab contaminants.
    ii. Cindy Auld and Kathy Miller – We typically do the full 8270 list at the SI stage and use detected/non-detect compounds to inform future sampling. We have limited site history, so non-PAH SVOCs may well be related to DoD site use. Transparency to the public is important. We need to run 8270 anyway so a) not cost savings and b) those compounds have been run and theoretically could be requested by the public. There is a public perception of risk if we haven't reported and evaluated that data. Labs run the full SVOC list anyway and just report what is requested so there is no reduction in price.
    iii. Harry Hendler - What DOD activities would have released SVOCs other than PAHs? E.g. what motor pool activities would have contributed other SVOCs?
    iv. Cindy Auld and Brad LaForest - Antifreeze (glycol), chlorinated hydrocarbons.
    v. Cindy Auld – Recommends non-fuel release areas run the full 8270 SVOC list.
    vi. Issue unresolved, see action items.
d. Soil depths - Some soil samples are listed as deep as 20 ft – this was estimated depth to bedrock, this maybe be shallower than 20 ft. Subsurface soil samples were removed from Small Arms Range area as USACE indicated that they don't expect metals to migrate vertically in soil at these types of sites.
e. Chlorinator building removed as the final discharge point – preceding samples will provide a better indication of potential contaminates in waste water.
f. Private well sampling could be increased beyond VOCs if samples on-site suggest sources of other contamination. VOCs will pick up certain indicators for VPH such as benzene.

6. Cindy Auld and Harry Hendler – Discussion of project action limits (PALs). HH screening values will use RSLs at target risk 1x10-6 and target HI of 0.1. State values may be considered if lower. Preferred not to assign surrogate values, but to identify gaps and evaluate if gaps are for potentially site-related compounds. Document should acknowledge PALs that can't be met. Tony and David to discuss and agree ecological benchmarks to use.
7. Joshua Mulvey informs group that he has not received responses from the six residential properties for rights of entry to inspect wells and sample groundwater. Stated he will follow up with another letter and a phone call if necessary. Harry Hendler states this may end up being a site visit to knock on doors and speak with homeowner's directly. Unresolved if

this would be a USACE activity or a Wood activity. Also agreed to defer private well sampling around remote Receiver Site location until after initial sampling is completed.

8. Brad LaForest and Kathy Miller – Discussion of sample FUDSChem naming conventions. Discussion on location ID and field sample ID nomenclature. Brad to provide Kathy with a location ID and field sample ID example for her review and approval.

Action Items:

Action	**Responsible Party**	**Due Date**
Revise Performance Work Statement and Modify Contract	Harry Hendler and Peter Baker	TBD
Revise Table 1 Summary of Analytical Program	Harry Hendler	24 January
Ecological Risk Assessment Offline Call/email	Cindy Auld, David Oster, Amy Quintin, and Tony Rodolakis	End of January
Revise and Confirm Analytical Program, Location IDs and Sample ID Nomenclature Table to Corps for Review	Brad LaForest and Kathy Miller	End of January

QAPP Worksheet #10: Conceptual Site Model

UFP-QAPP Manual Section 2.5.2
EPA 2106-G-05 Section 2.2.5

This worksheet describes an initial Conceptual Site Model (CSM) for the Charleston AFS. Although no soil and groundwater investigations have been conducted previously at the Site, there are basic physical and chemical processes that relate to potential migration of potential contaminants.

The CSM defines potential sources, migration and the exposure pathways and receptors, it evaluates whether exposure pathways are complete and informs human health and ecological risk assessments. Preparation of a CSM leads to identification of data gaps that may need to be filled to address uncertainty in risk assessments nature and extent of contamination needed to support a feasibility study.

Limited records are available relative to maintenance and waste practices conducted at the Charleston AFS. Solvent related compounds such as TCE were most likely used as a degreaser in automotive maintenance and painting. TCE and other solvents may have been used to clean electrical components and radar antennas. Various fuel products were also stored and used at various locations across the Site. Waste solvents and fuels may have been released directly to the ground surface or to subsurface soils though septic systems and floor drains or other discharge locations. Transformers containing PCBs used in the Hilltop Area were mounted on both concrete pads and poles and PCBs may have been released to the ground surface and/or the concrete pads. During the site visit in 2019, Wood observed a thick, black, oily substance at one of the transformer pads. It is unknown if these substances are currently sorbed to soils or concrete and have migrated to groundwater. Lead likely exists in shallow soils at the Small Arms Range, as lead bullet fragments have been found in this area.

The key components of the initial CSM are summarized below.

Overburden Soils

- Chlorinated solvents may have been used to clean tools and Radome equipment; waste solvents may have been released to soils following or during use.
- Fuels and oils were stored in USTs; improper use/disposal and leaks from these tanks may have released these substances to soils.
- Operations at the Small Arms Range most likely introduced lead to shallow soils.
- Contaminants may remain sorbed to fine grained soils and may partition to groundwater and air.
- Soils likely consist of weathered/reworked glacial till and local fill materials.
- Soils overlying bedrock are expected to be thin. Facility well construction near Route 15 suggests soils may be approximately 12 ft thick.
- Overburden soils in the Hilltop Area are thin to non-existent.

- Fill materials were placed in low areas between bedrock highs in the Hilltop Area to accommodate building structures and underground structures such as fuel tanks and septic fields.

Bedrock

- Due to thin soils, the volume of chlorinated solvents, fuels, and oils released to the ground surface and subsurface soils may have been sufficient to migrate to the bedrock surface and bedrock groundwater. Bedrock exposures are prevalent in the Hilltop Area.
- Bedrock consists of Devonian-Silurian Madrid Formation (MGS, 1985) described as calcareous sandstone and interbedded limestone.
- Bedrock also described locally as thickly bedded metasandstones (Westerman, 1983).
- Bedrock has high angle joints (dip angle > 75 degrees), which trend NW to SE, minor joints sets trend NE to SW (Westerman, 1983), bedrock is likely discretely fractured.
- Several bedrock exposures on the Hilltop appear well scoured by glacial action.

Overburden Groundwater

- Overburden groundwater occurs in response to areal recharge from precipitation, run-off, topographic controls, and to some degree by lateral or upward discharge from bedrock groundwater.
- Perched groundwater may exist locally in shallow till soils.
- Recharge water percolating through contaminated soils may leach, dissolve, contaminants to the water table.
- Water table is most likely shallow (<10 ft below ground surface [bgs]) if soils are of sufficient thickness to support an overburden system.
- Shallow overburden groundwater is present in the Northern Industrial area.
- Soils are not continuous in the Hilltop Area, overburden groundwater may be present seasonally in thin overburden.
- If present, groundwater flow from the Hilltop area is expected to be radial.
- Potentiometric surface most likely mimics topography.
- It is unknown if overburden groundwater is impacted by former Charleston AFS operations.

Bedrock Groundwater

- Bedrock groundwater occurs in response to areal recharge from precipitation, run-off, and topographic controls.
- Recharge water percolating through contaminated overburden and shallow bedrock materials may dissolve contaminants and migrate through the underlying fractured bedrock.
- Bedrock groundwater is most likely shallow (~10 to 15 ft bgs) in the Northern Industrial Area and beneath the Correctional Facility.
- Bedrock potentiometric surface most likely fluctuates widely in the Hilltop Area due to seasonal variations in recharge.
- Bedrock groundwater potentiometric surface is expected to mimic topography resulting in radial flow from the Hilltop Area.

- Due to bedrock structure, anisotropy may develop in a northwest to southeast orientation (Westerman, 1983) under a pumping stress.
- It is currently unknown if low level VOC contamination in the Facility Well is attributable to former Charleston AFS operations.

Surface Water

- Contaminants sorbed to surface soils may migrate overland with soil particles during heavy rain and snow melt events.
- Surface water runoff via overland flow is expected to occur during heavy rain events and thick snowpack melt.
- Three seep locations were mapped in October 2019, additional locations may occur during the Spring months (Wood, 2020b).
- USFWS National Wetland Inventory (Wood, 2020b) indicates no mapped wetlands on the Site.
- A palustrine scrub/shrub wetland exists along the northwestern portion of the Site (Wood, 2020b).
- Two potential vernal pools were identified near the radio transmitter/NCO club. No other vernal pools were identified on Site (Wood, 2020b).
- There are no perennial streams known on Site.
- Two storm water detention ponds currently exist on the Site and were constructed post Charleston AFS.
- Nearby streams include Rollins Brook ~½ mile to the southeast and Black Stream approximately 1 mile north and west of the Site.

Sediment

- Sediment likely collects in storm water detention structures that were constructed post closure of the Charleston AFS.
- Rollins Brook and Black Stream are not anticipated to have been affected by past Charleston AFS operations.
- Discharge of contaminated bedrock groundwater through overburden to Rollins Brook and Black Stream is unlikely to have impacted sediments if such discharge occurred.

QAPP Worksheet #11: Project/Data Quality Objectives

(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

DQOs were developed using United States Environmental Protection Agency (USEPA) *Guidance on Systematic Planning Using the Data Quality Objectives Process* USEPA QA/G-4 (USEPA, 2006).

1: State the Problem

The Site lacks data needed to draw conclusions regarding the presence or absence of chemical impacts to soil and groundwater from past disposal, maintenance, and material handling practices at the former Charleston AFS. Uncertainty exists with each potential source area, with the exception of the 3 USTs and soil that were removed from the Hilltop Area in 1989. There is a confirmed presence of TCE and cis-1,2-DCE in bedrock groundwater at the site, but the source is unknown. No other analytical chemical data exists for the Site.

The Sampling and Analysis Plan provides a list of potential source areas and summarizes the potentially affected media that will be evaluated during this phase of investigation.

2: Identify the Goals of the Study

The primary objective of this phase of the RI will involve sampling of soil and overburden groundwater to determine the presence/absence of contamination at potential source areas. Another objective will be to determine the presence/absence of contamination in the Mountain View Correctional Facility and residential water supply wells in the area. During this phase a third objective will be to determine on-Site background concentrations of SVOCs and metals in soil to allow comparison to soils data collected in impacted areas of the Site.

The goal of the data collection is to support a comparison against human health screening levels and ecological benchmarks, which will identify areas and chemical classes where more samples and analyses are needed (results above screening levels, and background if applicable). This QAPP provides the initial methodology necessary to complete the screening comparison, however data collected will be of sufficient quality to support quantitative risk assessment, if considered appropriate for inclusion at a further phase.

3: Identify Information Inputs

There have been no DoD soil and groundwater investigations completed at the former Charleston AFS. An inventory and removal action of transformers and some USTs was completed.

The New England District of the U.S. Army Corps of Engineers prepared an INPR for the Charleston AFS (D01ME011200) on 07 November 1988. The report which was approved on 20 January 1989, identified one DERP-FUDS project on the property. The project consisted of the testing and removal of twenty-two transformers and twenty-seven 275-gallon underground

heating fuel storage tanks as well as sampling the soil surrounding the tanks. The transformers and tanks were potential sources of environmental contaminants.

The New England District produced this Environmental Assessment to evaluate the environmental consequences of the removal action of three 10,000-gallon underground fuel storage tanks, twenty-one 275-gallon heating fuel USTs, two well house gasoline tanks, and twenty-two transformers from the Charleston AFS. A "Finding of No Significant Impact" was signed on 03 July 1989 for the proposed activity. The report determined the UST and transformer removal would have no impact on Federally listed, threatened or endangered species; any cultural resources present would have been heavily disturbed or destroyed during the construction of the UST, so no adverse effects were anticipated; and only temporary minimal impacts during the construction needed for the UST and transformer removal were expected Based on information on FUDSMIS, this project was completed in 1990, and in addition to the tank and transformer removal, the contractor removed 18 tons of petroleum-contaminated soil, 25 cubic yards of PCB contaminated soil, and 340 tons of waste oil-contaminated soil.

A PA Report was prepared for the Site by the USACE in January 2018.

Wood identified additional potential source areas following a review of historical air photography, existing site drawings and microfiche as-built drawings. This information and a summary of the findings are documented in Final Technical Memorandum Historic and Aerial Photography Research/Analysis (Wood, 2020a). The Technical Memorandum is provided in the SAP.

Wood conducted environmental, biological, and cultural resource surveys to identify protected natural and cultural resources at or near the Site that could be potentially impacted by RI field activities (Wood, 2020b).This information was used to develop sampling strategies presented in this QAPP.

The following data and informational needs for the site investigations are required to achieve the project goals:

- Soil boring advancement;
- Collection of soil, groundwater, drinking water and surface water samples; and
- Laboratory analysis of soil, groundwater, drinking water and surface water samples.

4: Define the Boundaries of the Study

Review of historical documents, site scoping visits, and post scoping conference calls have been and will continue to be used in determining the sampling locations. Sampling will be directed toward areas where contamination is suspected and surrounding areas.

5: Develop the Analytic Approach

Sampling of soil, groundwater, drinking water and surface water at potential source areas and along migration pathways is necessary to determine if contamination is present and whether human exposure exists. Various methods will be used to analyze the samples. If contamination is confirmed (see Step 2), then concentrations of those constituents will determine if further

actions (i.e., additional sampling, studies evaluation of remedial alternating and/or interim remedial actions) are necessary. **Worksheet #15** identifies: the PALs and method limits of quantitation (LOQ) for determining contamination presence/absence and quantitation in soil, groundwater, drinking water and surface water; the limits of detection (LOD) for determining with a high level of confidence (99%) that the analyte is absent; and, the method detection limits (MDLs) for determining with a high level of confidence (99%) that the analyte is present.

6: Specify Performance or Acceptance Criteria

The following performance and acceptance criteria will be used during remedial investigation activities:

- Daily personal protective equipment (PPE)/equipment checklist will be completed by the Wood Field Operations Lead. The Quality Assurance Manager will review and accept the final checklist.
- The Field Operations Lead will verify that field procedures defined in the QAPP are properly followed on a daily basis during field work. The Quality Assurance Manager or designee will verify field procedures are being conducted appropriately through field audits. Any deviations will be promptly addressed and documented.
- The laboratories, accredited under the DoD Environmental Laboratory Accreditation Program, will adhere to analytical performance/acceptance criteria, as detailed in the DoD Quality Systems Manual (QSM) V5.0 and defined on **Worksheets #12a and 12b**.
- Analytical methods will provide acceptable detection limits to confirm presence of contamination at concentrations defined in Step 5 and **Worksheet #15**.
- USEPA Stage 2B data verification will be conducted on 100 percent of the data and USEPA Stage 4 data validation will be conducted on 10 percent of the analytical data by an experienced chemist to assess the data usability. The data usability will then be evaluated by USACE for final approval. Data completeness of 90 percent usable data is required.
- The Remedial Investigation Report will be reviewed and accepted by USACE.

7: Develop the Detailed Plan for Obtaining Data

The basis for the design of the sampling program begins with an understanding of potential release areas, an understanding of the migration pathways, and potential receptors. The information in the CSM in **Worksheet #10** provide the basis for designing the sampling program.

The detailed sampling approach for the investigation is summarized in the QAPP worksheets. The information in **Worksheets #13, 14/16, 17, 18, and 20** is based on anticipated investigations to be conducted.

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: VOC SW8260C
Concentration Level: Low/Medium

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 20
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 20
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	-50% to +100%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)

(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Group or Method: SVOC 8270 SIM

Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 20
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 20
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	-50% to +100%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)

(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Group or Method: PCB SW8082A

Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 30[1]
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)

(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Group or Method: PCBs with Congeners SW8082A

Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	50-150%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	50-150%
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 30[1]
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	50-150%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: TAL Metals SW6020A/7471B
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ ½ LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 20
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 20[1]
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	30-120%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)

(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Group or Method: MA DEP VPH

Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	70-130%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 25
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	70-130%
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 30
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	70-130%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference
VPH = Volatile Petroleum Hydrocarbons

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: MA DEP EPH
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	40-140%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 25
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	40-140%
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 50 (Method limits)
Overall Precision	Field Duplicates	RPD ≤ 50
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	40-140%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
EPH = Extractable Petroleum Hydrocarbons
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: pH SW9045D
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	±0.1 pH units
Overall Precision	Field Duplicates	RPD ≤ 30

Notes:
LCS = Laboratory Control Sample
QC = Quality Control
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Grain Size ASTM D422
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
NA	NA	NA

Notes: NA = Not Applicable

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: FOC Lloyd Kahn
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	80-120%
Overall Precision	Field Duplicates	RPD ≤ 30

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
FOC = Fractional Organic Carbon
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Waste Characterization TCLP VOC SW1311/8260C
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	-50% to +100%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
TCLP = Toxicity Characteristic Leaching Procedure
VOC = Volatile Organic Compounds

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Waste Characterization TCLP SVOC SW1311/8270D
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	-50% to +100%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
SVOC = Semi volatile Organic Compounds
TCLP = Toxicity Characteristic Leaching Procedure

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Waste Characterization TCLP Pesticides SW1311/8081B
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
TCLP = Toxicity Characteristic Leaching Procedure

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Waste Characterization TCLP Herbicides SW1311/8151A
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
TCLP = Toxicity Characteristic Leaching Procedure

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)
Matrix: Soil
Analytical Group or Method: Waste Characterization PCBs SW8082A
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
PCBs = Polychlorinated Biphenyls
QC = Quality Control
QSM = Quality Systems Manual

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Waste Characterization TCLP Metals SW1311/6010C/7470A
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ ½ LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
TCLP = Toxicity Characteristic Leaching Procedure

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Reactive Cyanide Chapter Seven, 7.3.3.2
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ ½ LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	80-120%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Reactive Sulfide Chapter Seven, 7.3.4.2
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	80-120%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Ignitability SW1010MOD
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	27°C ± 2°C

Notes:
LCS = Laboratory Control Sample
QC = Quality Control

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Water
Analytical Group or Method: VOC SW8260C
Concentration Level: Low

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 20
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 20
Overall Precision	Field Duplicates	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits
Analytical Accuracy/Bias (laboratory)	Internal Standard Recoveries	-50% to +100%[1]

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference
VOC = Volatile Organic Compounds

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Water
Analytical Group or Method: PCBs SW8082A
Concentration Level: Low

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	QSM Appendix C limits
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	QSM Appendix C limits
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 30[1]
Overall Precision	Field Duplicates	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MS = Matrix Spike
MSD = Matrix Spike Duplicate
PCBs = Polychlorinated Biphenyls
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Water
Analytical Group or Method: MA DEP VPH
Concentration Level: Low

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	70-130%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 25
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	70-130%
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 30[1]
Overall Precision	Field Duplicates	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	70-130%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference
VPH = Volatile Petroleum Hydrocarbons

QAPP Worksheet #12a: Katahdin Analytical Services Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Water
Analytical Group or Method: MA DEP EPH
Concentration Level: Low

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Overall accuracy/Bias (contamination)	QC Blanks	No target analyte concentrations ≥ 1/2 LOQ[1]
Analytical Accuracy (laboratory)	Laboratory Control Sample (LCS)	40-140%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicate (LCSD)	RPD ≤ 25
Analytical Accuracy/Bias (matrix interference)	Matrix Spike (MS)	40-140%
Analytical Precision (matrix interference)	Matrix Spike Duplicates (MSD)	RPD ≤ 25[1] (Method Limits)
Overall Precision	Field Duplicates	RPD ≤ 30
Analytical Accuracy/Bias (matrix interference)	Surrogate Recoveries	40-140%

Notes: [1] Acceptance criteria set in accordance with DoD QSM 5.3
EPH = Extractable Petroleum Hydrocarbons
LCS = Laboratory Control Sample
LCSD = Laboratory Control Sample Duplicate
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MS = Matrix Spike
MSD = Matrix Spike Duplicate
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference

QAPP Worksheet #12b: Eurofins TestAmerica Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Gross Alpha & Gross Beta SW9310
Concentration Level: None

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
Analytical Accuracy (laboratory)	Lab Control Sample (LCS)	75 - 125%

Notes: [1]
LCS = Laboratory Control Sample

QAPP Worksheet #12b: Eurofins TestAmerica Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: Gamma Spec by Separation Resins GA-01-R
Concentration Level: N/A

Data Quality Indicator (DQI)	Quality control (QC) sample or measurement performance activity	Measurement Performance Criteria
NA	NA	NA

Notes:

QAPP Worksheet #13: Secondary Data Uses and Limitations

(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements For Evaluating Existing Data)

Data type	Source(s)	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Meteorological	National Weather Service	Planning field investigations Estimating infiltration and seasonal fluctuations in storm water runoff	Weather reports published daily/hourly. No known limitations. Published data are available for past 20 years. No known limitations.
Topographic	USGS and NOAA	Elevation data for field investigation and remedial investigation report https://coast.noaa.gov/dataviewer/#/lidar/search/ Lineament analysis Surface water drainage pathways	No known limitations.
Hydrogeologic and Geologic	USGS, Maine DEP, and Maine Geological Survey	Location of significant aquifers and faults Well yields Surficial geology types and rock formations, fracture orientations	No known limitations. Some well driller supplied information may not be accurate in State or Federal databases.
Potential Release Areas	Interviews; Records Reviews	Focuses investigation media and locations; contributes to understanding of CSM and potential fate/transport of contamination	Recollection of interviewees may be uncertain; records may not be available for all releases to all media; some information may be of unknown quality.
Past Site Uses	Plant operating records and personnel interviews	Focuses investigation media and locations; contributes to understanding of CSM and potential fate/transport of contamination	Records for operations prior may not be readily available or limited in scope.

QAPP Worksheet #14/16: Project Tasks & Schedule

(UFP-QAPP Manual Section 2.8.2)
(EPA 2106-G-05 Section 2.2.4)

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Mobilization/demobilization	Wood	Late Summer 2020	Summer 2021	None	None
Soil boring advancement/abandonment	Wood	Fall 2020	Late Fall 2020	Field notes and boring logs (included in Remedial Investigation Report)	Draft RI Fall 2021
Sample collection- Surface soils	Wood	Fall 2020	Late Fall 2020	Field notes and soil sample collection log (included in Remedial Investigation Report)	Draft RI Fall 2021
Sample collection- Subsurface soils	Wood	Fall 2020	Late Fall 2020	Field notes and soil sample collection log (included in Remedial Investigation Report)	Draft RI Fall 2021
Sample collection- groundwater	Wood	Fall 2020	Late Fall 2020	Field notes and groundwater sample collection log (included in Remedial Investigation Report)	Draft RI Fall 2021
Sample collection- groundwater from private and public drinking water wells	Wood	Fall 2020	Summer 2021	Field notes and groundwater sample collection log (included in Remedial Investigation Report)	Draft RI Fall 2021

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Sample collection- surface water	Wood	Fall 2020	Summer 2021	Field notes and surface water collection log (included in Remedial Investigation Report)	Draft RI Fall 2021
Analysis	Katahdin	Fall 2020	Late Fall 2020 to Summer 2021	Report of Analyses/Data package (included in Remedial Investigation Report)	Draft RI Fall 2021
Validation	Wood	Winter 2021	Summer 2021	Validation Summary (included in Remedial Investigation Report)	Draft RI Fall 2021
FUDSChem Data Submittal	Wood	Fall 2020	Fall 2021	FUDSChem	90 days after Sampling Completed
Usability assessment	Wood	Spring 2021	Fall 2021	Meeting minutes/Usability assessment summary report	Spring 2021
Remedial Investigation Report	Wood	Fall 2021 Draft	Spring 2022 Final	Remedial Investigation Report	Spring 2022

Project Schedule

The field work is anticipated to be performed in the late summer and fall of 2020. The scope of work includes quarterly sampling (4 rounds) of seeps, Correctional Facility and residential water supply wells; therefore field work will not be completed until June 2020. The Draft RI report is anticipated to be issued in the fall of 2021. A detailed field activities schedule will be provided in Final SAP. The general overall schedule is shown below.

- Draft SAP and QAPP: March 31, 2020
- Draft Final SAP and QAPP: May 29, 2020

- Final SAP and QAPP: July 31, 2020
- Mobilization Activities: August 3 - August 28, 2020
- Field Work: September 1, 2020 – June 2021
- Draft RI Report: November 2021
- Draft Final RI Report: January 2022
- Final RI Report: March 2022

QAPP Worksheet #15a: Katahdin Analytical Services Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)

(EPA 2106-G-05 Section 2.2.6)

Detections limits established for this project are consistent with low concentration methods used during Remedial Investigations under CERCLA to test for a broad range of analytes. These methods have detection limits similar to routine USEPA Contract Laboratory Program (CLP) methods and are designed to provide information on the presence of contamination for a large suite of analytes from multiple methods. Analytical methods include target compounds/analytes identified in the DOD QSM with MDLs, LODs, and LOQs that are typical of low concentration methods and the USEPA Target Compound List (TCL) and Target Analyte List (TAL).

In accordance with CERCLA guidelines, the RSLs and benchmark criteria will be used to screen analytical data and determine which parameters will be carried through the RI and ultimately a quantitative risk evaluation. It is not a project objective of the Remedial Investigation to obtain MDLs below PALs for all target analytes. Obtaining very low MDLs for a full suite of analytes is not needed to complete this phase of the Remedial Investigation at the Site. Systematic investigations at several locations are designed to test for a wider list of parameters including VOCs, SVOCs, PCBs, and metals. The USEPA RSLs and benchmark criteria referenced in Worksheet 15 are very conservative and are used only as screening values in the evaluation of analytes detected in Site samples. Project-specific risk assessments will be completed based on factors related to the contamination assessment conceptual model and risk factors identified at the Site.

Human health and ecological PALs listed on Worksheet 15 are based on USEPA Regional Screening Levels (RSLs) and other various benchmark criteria. In some cases, the RSLs and benchmark criteria are set at very low concentrations that are lower than the PALs established for the analytical methods.

PALs for EPH and VPH analyses are based on Remediation Guidelines for Petroleum Contaminated Sites in Maine (MEDEP, 2014) and are for the purpose of comparison only as EPH and VPH are non CERCLA compounds.

There are no applicable screening levels for parameters that do not have a PAL value.

The MDLs have been evaluated in comparison to the risk-based screening levels to ensure they are sufficiently low. All MDLs are below ecological benchmarks. The majority of MDLs are also below the RSLs for human health. Exceptions are generally within an acceptable margin that avoids masking potential risk drivers, with the exception of 1,2-Dibromo-3-chloropropane. The results for all parameters listed below will be reviewed in the context of detected soil concentrations and related parameters to ensure that there are no significant data gaps for expected site-related concentrations. Details related to MDLs exceeding PALs are as follows:

- Eight MDLs for VOCs in water are not sufficiently low. In general data collection in aqueous media for potential VOCs is sufficient to capture potential risk drivers and avoid masking low detected results that may impact future actions. Further details are provided below for specific VOCs:

    - Five VOC PALs are within one order of magnitude of the MDL (1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane1,2-dichloroethane, bromodichloromethane, and chloroform). Therefore, all potential risk drivers will be detected, as the typical remediation decision threshold is one order of magnitude above the RSL for non-cancer endpoints and two orders of magnitude above the RSL based on a cancer endpoint. Any concentrations present below the detection limit are below potential risk thresholds that would impact future action.

    - Two VOC PALs are between 1 and 2 orders of magnitude lower than the MDL (1,2-Dibromoethane (EDB) and vinyl chloride). The PALs are based on RSLs, which are based on a cancer endpoint at a target excess lifetime cancer risk (ELCR) of $1x10^{-6}$. The non-cancer based RSL is more than two orders of magnitude higher than the cancer based RSL for these two parameters. Therefore, as the cancer-based RSL is based on an ELCR of $1x10^{-6}$ and the typical remediation decision threshold for carcinogens is two orders of magnitude above the RSL based on a cancer endpoint, all potential risk drivers will be captured for these two parameters. Any concentrations present below the MDL are below potential risk thresholds that would impact future action.

    - One VOC PAL is over 2 orders of magnitude lower than the MDL (1,2-Dibromo-3-chloropropane). This PAL is also based on an RSL, which is based on a cancer endpoint at an ELCR of $1x10^{-6}$. The non-cancer based RSL is more than two orders of magnitude higher than the cancer based RSL for this parameter. This MDL has the potential to miss a risk driver, however this parameter is not specifically anticipated to be site-related (i.e., associated with agricultural use), and therefore is unlikely to be a risk-driver for the site. When results are obtained, any related detected compounds including whether this parameter is detected in soil will be evaluated to identify whether this chemical is likely to be present, and therefore would be expected to be a significant data gap.

- MDLs for PCBs in water are not sufficiently low for several Aroclors, however this difference is less than one order of magnitude (Aroclor 1221, Aroclor 1232, Aroclor 1242, Aroclor 1248, Aroclor 1254, and Aroclor 1260). As PCBs in soil are a primary target analyte class for the RI, there will be sufficient data available to evaluate whether potential non-detects could be masking low detected results in specific areas. Additionally, the Tapwater RSLs are a conservative estimate to evaluate groundwater seeps, which are not expected to be a source of drinking water.

- Two MDLs for PCBs congeners in soil are not sufficiently low (3,3',4,4',5,5'-Hexachlorobiphenyl and 3,3',4,4',5-Pentachlorobiphenyl). The difference is less than an order of magnitude, and the PAL is based on the RSL, suggesting the likelihood that low concentrations below the detection limit could mask a risk driver is minimal. Additionally, the MDLs for aroclors in soil are sufficiently low, and PCB congeners are primarily collected for comparison to ecological benchmarks based on 2,3,7,8-TCDD. Therefore, the impact is considered negligible.

Parameters discussed with PALs above the MDL are highlighted in this worksheet. Parameters with PALs below the Project Quantitation Limit Goal are also highlighted. If the PAL is less than the PQLG but greater than the MDL, a detection between the LOQ and MDL will be J flagged. Therefore, the additional highlighted parameters (e.g. trichloroethene) are not discussed further.

PCBs will be assessed as outlined in *Framework for Application of the Toxicity Equivalence Methodology for Polychlorinated Dioxins, Furans, and Biphenyls in Ecological Risk Assessment* (USEPA, 2008). In this approach, congeners will be the principal line of evidence, as these are individual compounds, whereas Aroclors are mixtures of compounds which would have degraded over time. The congener analysis is performed by multiplying each congener concentration by a toxicity equivalence factor (TEF) so that toxicity of each congener is expressed in terms of 2,3,7,8-TCDD. 2,3,7,8-TCDD is most toxic of the dioxin/PCB congeners. Most of the congeners of interest are approximately three orders of magnitude less toxic than TCDD (i.e., a TEF of 0.0001 or 0.0003). The TEF-adjusted congener concentrations are then summed to calculate a toxicity equivalence quotient (TEQ) which expresses all the congener concentrations as a single soil concentration in terms of 2,3,7,8-TCDD. This TEQ concentration is then compared to an ecological soil screening benchmark for 2,3,7,8-TCDD. Where a given congener is not detected, the 1/2 reporting limit will be used in the calculation. As part of the Uncertainty Discussion, a brief quantitative sensitivity analysis can be performed by using the full detection limits instead. However, because the TEFs are so low, congeners would need to be detected at tens to hundreds of times above the PAL in order to contribute any meaningful level of toxicity. In other words, congener MDLs above PALs are not anticipated to be a significant issue.

Blue shading indicates the LOQ was greater than the FUDSChem calculated PQLG and the PQLG has been set at the LOQ.

Yellow shading indicates the MDL and/or LOQ is greater than the PAL.

Red shading indicates the PAL is less than the PQLG.

Matrix: Aqueous
Analytical Method: MA DEP EPH
Concentration level (if applicable): N/A Units: µg/L

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
C11-C22 Petroleum Hydrocarbons, Aromatic	200	Charleston Petroleum Screening Aqueous	100	100	75.0	50.0
C19-C36 Petroleum Hydrocarbons, Aliphatic	8000	Charleston Petroleum Screening Aqueous	2670	100	75.0	50.0
C9-C18 Petroleum Hydrocarbons, Aliphatic	500	Charleston Petroleum Screening Aqueous	167	100	75.0	50.0

Notes:
EPH = Extractable Petroleum Hydrocarbons
LOD = Limit of Detection
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MDL = Method Detection Limit
µg/L = micrograms per liter

Matrix: Aqueous
Analytical Method: MA DEP VPH
Concentration level (if applicable): N/A Units: µg/L

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
C5-C8 Volatile Petroleum Hydrocarbons, Aliphatic	200	Charleston Petroleum Screening Aqueous	100	100	75.0	50.0
C9-C10 Volatile Petroleum Hydrocarbons, Aromatic	200	Charleston Petroleum Screening Aqueous	100	100	75.0	50.0
C9-C12 Volatile Petroleum Hydrocarbons, Aliphatic	500	Charleston Petroleum Screening Aqueous	167	100	75.0	50.0

Notes:

LOD = Limit of Detection

LOQ = Limit of Quantitation

MA DEP = Massachusetts Department of Environmental Protection

MDL = Method Detection Limit

µg/L = micrograms per liter

VPH = Volatile Petroleum Hydrocarbons

Matrix: Aqueous
Analytical Method: PCB SW8082A
Concentration level (if applicable): N/A Units: µg/L

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
PCB-1016 (Aroclor 1016)	0.140	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.0150
PCB-1221 (Aroclor 1221)	0.00470	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.0200
PCB-1232 (Aroclor 1232)	0.00470	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.00800
PCB-1242 (Aroclor 1242)	0.00780	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.0180
PCB-1248 (Aroclor 1248)	0.00780	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.0200
PCB-1254 (Aroclor 1254)	0.00780	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.00800
PCB-1260 (Aroclor 1260)	0.00780	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.0170
PCB-1262 (Aroclor 1262)	0.0440	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.00700
PCB-1268 (Aroclor 1268)	0.0440	Charleston Human Health Residential Screening Aqueous	0.0500	0.0500	0.0250	0.00700

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
PCBs = Polychlorinated Biphenyls
µg/L = micrograms per liter

Matrix: Aqueous
Analytical Method: VOC SW8260C
Concentration level (if applicable): Low

Units: µg/L

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
1,1,1-Trichloroethane	76.0	Charleston Eco Screening Liquid	25.3	1.00	0.500	0.200
1,1,2,2-Tetrachloroethane	0.0760	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.380
1,1,2-Trichloro-1,2,2-trifluoroethane	1000	Charleston Human Health Residential Screening Aqueous	333	1.00	0.500	0.310
1,1,2-Trichloroethane	0.0410	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.330
1,1-Dichloroethane	2.80	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.210
1,1-Dichloroethene	28.0	Charleston Human Health Residential Screening Aqueous	9.33	1.00	0.500	0.350
1,2,3-Trichlorobenzene	0.700	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.270
1,2,4-Trichlorobenzene	0.400	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.370
1,2-Dibromo-3-chloropropane	0.000330	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.750	0.500

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
1,2-Dibromoethane (EDB)	0.00750	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.220
1,2-Dichlorobenzene	23.0	Charleston Eco Screening Liquid	7.67	1.00	0.500	0.150
1,2-Dichloroethane	0.170	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.200
1,2-Dichloropropane	0.820	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.250
1,3-Dichlorobenzene	22.0	Charleston Eco Screening Liquid	7.33	1.00	0.500	0.260
1,4-Dichlorobenzene	0.480	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.240
2-Butanone (MEK)	560	Charleston Human Health Residential Screening Aqueous	187	5.00	2.50	1.31
2-Hexanone	3.80	Charleston Human Health Residential Screening Aqueous	5.00	5.00	2.50	1.70
4-Methyl-2-pentanone (MIBK)	170	Charleston Eco Screening Liquid	56.7	5.00	2.50	1.32
Acetone	1400	Charleston Human Health Residential Screening Aqueous	467	5.00	2.50	2.21
Benzene	0.460	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.260

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Bromochloromethane	8.30	Charleston Human Health Residential Screening Aqueous	2.77	1.00	0.500	0.210
Bromodichloromethane	0.130	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.330
Bromoform	3.30	Charleston Human Health Residential Screening Aqueous	1.10	1.00	0.500	0.230
Bromomethane	0.750	Charleston Human Health Residential Screening Aqueous	2.00	2.00	1.00	0.490
Carbon disulfide	0.920	Charleston Eco Screening Liquid	1.00	1.00	0.500	0.250
Carbon tetrachloride	0.460	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.220
Chlorobenzene	7.80	Charleston Human Health Residential Screening Aqueous	2.60	1.00	0.500	0.220
Chloroethane	2100	Charleston Human Health Residential Screening Aqueous	700	2.00	1.00	0.550
Chloroform	0.220	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.320
Chloromethane	19.0	Charleston Human Health Residential Screening Aqueous	6.33	2.00	1.00	0.360

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
cis-1,2-Dichloroethene	3.60	Charleston Human Health Residential Screening Aqueous	1.20	1.00	0.500	0.210
cis-1,3-Dichloropropene	N/A		1.00	1.00	0.500	0.190
Cyclohexane	1300	Charleston Human Health Residential Screening Aqueous	433	1.00	0.500	0.310
Dibromochloromethane	0.870	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.300
Dichlorodifluoromethane	20.0	Charleston Human Health Residential Screening Aqueous	6.67	2.00	1.00	0.240
Ethylbenzene	1.50	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.210
Isopropylbenzene (Cumene)	2.60	Charleston Eco Screening Liquid	1.00	1.00	0.500	0.230
m,p-Xylene	19.0	Charleston Human Health Residential Screening Aqueous	6.33	2.00	1.00	0.590
Methyl acetate	2000	Charleston Human Health Residential Screening Aqueous	667	1.00	0.750	0.530
Methyl tert-butyl ether (MTBE)	14.0	Charleston Human Health Residential Screening Aqueous	4.67	1.00	0.500	0.360
Methylcyclohexane	N/A		1.00	1.00	0.500	0.300

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Methylene chloride	11.0	Charleston Human Health Residential Screening Aqueous	5.00	5.00	2.50	1.13
o-Xylene	19.0	Charleston Human Health Residential Screening Aqueous	6.33	1.00	0.500	0.250
Styrene	32.0	Charleston Eco Screening Liquid	10.7	1.00	0.500	0.230
Tetrachloroethene (PCE)	4.10	Charleston Human Health Residential Screening Aqueous	1.37	1.00	0.500	0.400
Toluene	62.0	Charleston Eco Screening Liquid	20.7	1.00	0.500	0.270
trans-1,2-Dichloroethene	36.0	Charleston Human Health Residential Screening Aqueous	12.0	1.00	0.500	0.250
trans-1,3-Dichloropropene	N/A		1.00	1.00	0.500	0.200
Trichloroethene (TCE)	0.280	Charleston Human Health Residential Screening Aqueous	1.00	1.00	0.500	0.280
Trichlorofluoromethane	520	Charleston Human Health Residential Screening Aqueous	173	2.00	1.00	0.240
Vinyl chloride	0.0190	Charleston Human Health Residential Screening Aqueous	2.00	2.00	1.00	0.250

Notes:

LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
µg/L = micrograms per liter
N/A = Not Applicable
VOC = Volatile Organic Compounds

Matrix: Solid
Analytical Method: SVOC SW8270D SIM
Concentration level (if applicable): N/A

Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
1-Methylnaphthalene	0.140	Charleston Eco Screening Solid	0.0467	0.0200	0.0100	0.00167
2,3,4,6-Tetrachlorophenol	0.199	Charleston Eco Screening Solid	0.100	0.100	0.0500	0.00270
2,4,5-Trichlorophenol	14.1	Charleston Eco Screening Solid	4.70	0.100	0.0500	0.00254
2,4,6-Trichlorophenol	6.30	Charleston Human Health Residential Screening Solid	2.10	0.100	0.0500	0.00325
2,4-Dichlorophenol	19.0	Charleston Human Health Residential Screening Solid	6.33	0.0200	0.0100	0.00218
2,4-Dimethylphenol	0.0100	Charleston Eco Screening Solid	0.0200	0.0200	0.0100	0.00324
2,4-Dinitrophenol	0.0609	Charleston Eco Screening Solid	0.100	0.100	0.0700	0.0600
2-Chlorophenol	0.390	Charleston Eco Screening Solid	0.130	0.100	0.0500	0.00532
2-Methylnaphthalene	3.60	Charleston Petroleum Screening Solid	1.20	0.0200	0.0100	0.00221
2-Methylphenol (o-Cresol)	0.670	Charleston Eco Screening Solid	0.223	0.100	0.0500	0.00853
2-Nitrophenol	1.60	Charleston Eco Screening Solid	0.533	0.100	0.0500	0.0100
4,6-Dinitro-2-methylphenol	0.144	Charleston Eco Screening Solid	0.200	0.200	0.100	0.0200
4-Chloro-3-methylphenol	7.95	Charleston Eco Screening Solid	2.65	0.100	0.0500	0.00433
4-Nitrophenol	5.12	Charleston Eco Screening Solid	1.70	0.100	0.0500	0.00798
Acenaphthene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00145

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Acenaphthylene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00122
Anthracene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00124
Benzo(a)anthracene	1.10	Charleston Human Health Residential Screening Solid	0.367	0.0200	0.0100	0.00193
Benzo(a)pyrene	0.110	Charleston Human Health Residential Screening Solid	0.0367	0.0200	0.0100	0.00329
Benzo(b)fluoranthene	1.10	Charleston Human Health Residential Screening Solid	0.367	0.0200	0.0100	0.00244
Benzo(g,h,i)perylene	1.10	Charleston Eco Screening Solid	0.367	0.0200	0.0100	0.00195
Benzo(k)fluoranthene	1.10	Charleston Eco Screening Solid	0.367	0.0200	0.0100	0.00310
Benzoic acid	1.00	Charleston Eco Screening Solid	0.333	0.0400	0.0200	0.00510
Carbazole	79.0	Charleston Eco Screening Solid	26.3	0.0200	0.0100	0.00131
Chrysene	1.10	Charleston Eco Screening Solid	0.367	0.0200	0.0100	0.00173
Cresols, m- & p-	163	Charleston Eco Screening Solid	54.3	0.100	0.0500	0.00991
Dibenz(a,h)anthracene	0.110	Charleston Human Health Residential Screening Solid	0.0367	0.0200	0.0100	0.00184
Dibenzofuran	6.10	Charleston Eco Screening Solid	2.03	0.0200	0.0100	0.00191
Fluoranthene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00176
Fluorene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00315
Indeno(1,2,3-c,d)pyrene	1.10	Charleston Eco Screening Solid	0.367	0.0200	0.0100	0.00185
Naphthalene	1.70	Charleston Petroleum Screening Solid	0.567	0.0200	0.0100	0.00257

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Pentachlorophenol	1.00	Charleston Human Health Residential Screening Solid	0.333	0.100	0.0500	0.0100
Phenanthrene	29.0	Charleston Eco Screening Solid	9.67	0.0200	0.0100	0.00177
Phenol	0.790	Charleston Eco Screening Solid	0.263	0.100	0.0500	0.00607
Pyrene	1.10	Charleston Eco Screening Solid	0.367	0.0200	0.0100	0.00210

Notes:

LOD = Limit of Detection

LOQ = Limit of Quantitation

MDL = Method Detection Limit

mg/kg = milligram per kilogram

SIM = Selective Ion Monitoring

SVOC = Semi volatile Organic Compound

Matrix: Solid
Analytical Method: FOC Lloyd Kahn
Concentration level (if applicable): N/A Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Fractional Organic Carbon	N/A		0.000400	0.000400	0.000200	0.000100

Notes:
FOC = Fractional Organic Carbon
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram
N/A = Not Applicable

Matrix: Solid
Analytical Method: MA DEP EPH
Concentration level (if applicable): N/A Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
C11-C22 Petroleum Hydrocarbons, Aromatic	460	Charleston Petroleum Screening Solid	153	20.0	15.0	10.0
C19-C36 Petroleum Hydrocarbons, Aliphatic	10000	Charleston Petroleum Screening Solid	3330	20.0	15.0	10.0
C9-C18 Petroleum Hydrocarbons, Aliphatic	2700	Charleston Petroleum Screening Solid	900	20.0	15.0	10.0

Notes:
EPH = Extractable Petroleum Hydrocarbons
LOD = Limit of Detection
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MDL = Method Detection Limit
mg/kg = milligram per kilogram

Matrix: Solid
Analytical Method: MA DEP VPH
Concentration level (if applicable): N/A Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
C5-C8 Volatile Petroleum Hydrocarbons, Aliphatic	1400	Charleston Petroleum Screening Solid	467	25.0	19.0	12.5
C9-C10 Volatile Petroleum Hydrocarbons, Aromatic	75	Charleston Petroleum Screening Solid	25.0	25.0	19.0	12.5
C9-C12 Volatile Petroleum Hydrocarbons, Aliphatic	2700	Charleston Petroleum Screening Solid	900	25.0	19.0	12.5

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MA DEP = Massachusetts Department of Environmental Protection
MDL = Method Detection Limit
mg/kg = milligram per kilogram
VPH = Volatile Petroleum Hydrocarbons

Matrix: Solid
Analytical Method: Trace Metals SW6020A
Concentration level (if applicable): N/A

Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Aluminum	7700	Charleston Human Health Residential Screening Solid	2570	30.0	4.00	0.510
Antimony	0.270	Charleston Eco Screening Solid	0.100	0.100	0.0500	0.0200
Arsenic	0.680	Charleston Human Health Residential Screening Solid	0.500	0.500	0.400	0.150
Barium	330	Charleston Eco Screening Solid	110	0.200	0.100	0.0300
Beryllium	16.0	Charleston Human Health Residential Screening Solid	5.33	0.100	0.0200	0.00410
Cadmium	0.360	Charleston Eco Screening Solid	0.120	0.100	0.0200	0.00760
Calcium	N/A		10.0	10.0	8.00	3.83
Chromium	26.0	Charleston Eco Screening Solid	8.67	0.400	0.300	0.0500
Cobalt	2.30	Charleston Human Health Residential Screening Solid	0.767	0.100	0.0300	0.00540
Copper	28.0	Charleston Eco Screening Solid	9.33	0.300	0.200	0.0700
Iron	5500	Charleston Human Health Residential Screening Solid	1830	30.0	4.00	0.510
Lead	11.0	Charleston Eco Screening Solid	3.67	0.100	0.0500	0.0200
Magnesium	N/A		0.500	0.500	0.400	0.150
Manganese	180	Charleston Human Health Residential Screening Solid	60.0	0.200	0.100	0.0300
Nickel	38.0	Charleston Eco Screening Solid	12.7	0.100	0.0200	0.00760
Potassium	N/A		10.0	10.0	8.00	3.83
Selenium	0.520	Charleston Eco Screening Solid	0.400	0.400	0.300	0.0500
Silver	4.20	Charleston Eco Screening Solid	1.39	0.100	0.0300	0.00540
Sodium	N/A		30.0	30.0	4.00	0.510

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Thallium	0.0500	Charleston Eco Screening Solid	0.100	0.100	0.0500	0.0200
Vanadium	7.80	Charleston Eco Screening Solid	2.60	0.500	0.400	0.150
Zinc	46.0	Charleston Eco Screening Solid	15.3	0.200	0.100	0.0300

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram
N/A = Not Applicable

Matrix: Solid
Analytical Method: Mercury SW7471B
Concentration level (if applicable): N/A

Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Mercury	0.0130	Charleston Eco Screening Solid	0.0300	0.0300	0.0170	0.00520

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram

Matrix: Solid
Analytical Method: PCB Aroclors SW8082A
Concentration level (if applicable): N/A

Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
PCB-1016 (Aroclor 1016)	0.410	Charleston Human Health Residential Screening Solid	0.137	0.0100	0.00850	0.00600
PCB-1221 (Aroclor 1221)	0.200	Charleston Human Health Residential Screening Solid	0.170	0.170	0.00850	0.00790
PCB-1232 (Aroclor 1232)	0.170	Charleston Human Health Residential Screening Solid	0.170	0.170	0.00850	0.00930
PCB-1242 (Aroclor 1242)	0.0410	Charleston Eco Screening Solid	0.170	0.170	0.00850	0.00580
PCB-1248 (Aroclor 1248)	0.00730	Charleston Eco Screening Solid	0.170	0.170	0.00850	0.00610
PCB-1254 (Aroclor 1254)	0.0410	Charleston Eco Screening Solid	0.0137	0.0100	0.00850	0.00470
PCB-1260 (Aroclor 1260)	0.240	Charleston Human Health Residential Screening Solid	0.0800	0.0100	0.00850	0.00600
PCB-1262 (Aroclor 1262)	0.230	Charleston Human Health Residential Screening Solid	0.170	0.170	0.00850	0.00370
PCB-1268 (Aroclor 1268)	0.230	Charleston Human Health Residential Screening Solid	0.170	0.170	0.00850	0.00260

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram
PCB = Polychlorinated Biphenyl

Matrix: Solid
Analytical Method: PCB Congeners SW8082A
Concentration level (if applicable): N/A

Units: mg/kg

Analyte	Project Action Limit*	Project Action Limit Reference*	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
2,3,3',4,4',5,5'-Heptachlorobiphenyl	0.130	Charleston Human Health Residential Screening Solid	0.0433	0.00100	0.000500	0.0000690
2,3,3',4,4',5'-Hexachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00100	0.000500	0.0000800
2,3,3',4,4',5-Hexachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00100	0.000500	0.000110
2,3,3',4,4'-Pentachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00200	0.00100	0.000820
2,3',4,4',5,5'-Hexachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.0100	0.00500	0.0000580
2,3,4,4',5-Pentachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00100	0.000500	0.0000670
2,3',4,4',5'-Pentachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00100	0.000500	0.0000670
2,3',4,4',5-Pentachlorobiphenyl	0.120	Charleston Human Health Residential Screening Solid	0.0400	0.00100	0.000500	0.00000900
3,3',4,4',5,5'-Hexachlorobiphenyl	0.000120	Charleston Human Health Residential Screening Solid	0.00100	0.00100	0.000500	0.000130
3,3',4,4',5-Pentachlorobiphenyl	0.0000360	Charleston Human Health Residential Screening Solid	0.00100	0.00100	0.000500	0.000160
3,3',4,4'-Tetrachlorobiphenyl	0.0380	Charleston Human Health Residential Screening Solid	0.0127	0.00100	0.000500	0.000200
3,4,4',5-Tetrachlorobiphenyl	0.0120	Charleston Human Health Residential Screening Solid	0.00400	0.00100	0.000500	0.000250

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram
PCB = Polychlorinated Biphenyl
*PCB Congeners will be assessed as 2,3,7,8-TCDD using the Toxicity Equivalency (TEQ) Method (USEPA, 2008).

Matrix: Solid
Analytical Method: VOC SW8260C
Concentration level (if applicable): Low/Medium Units: mg/kg

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
1,1,1-Trichloroethane	260	Charleston Eco Screening Solid	86.7	0.00500	0.00250	0.000420
1,1,2,2-Tetrachloroethane	0.127	Charleston Eco Screening Solid	0.0423	0.00500	0.00250	0.000840
1,1,2-Trichloro-1,2,2-trifluoroethane	670	Charleston Human Health Residential Screening Solid	223	0.00500	0.00250	0.000900
1,1,2-Trichloroethane	0.150	Charleston Human Health Residential Screening Solid	0.0500	0.00500	0.00250	0.000970
1,1-Dichloroethane	3.60	Charleston Human Health Residential Screening Solid	1.20	0.00500	0.00250	0.00170
1,1-Dichloroethene	11.0	Charleston Eco Screening Solid	3.67	0.00500	0.00250	0.000930
1,2,3-Trichlorobenzene	6.30	Charleston Human Health Residential Screening Solid	2.10	0.00500	0.00250	0.000760
1,2,4-Trichlorobenzene	0.270	Charleston Eco Screening Solid	0.0900	0.00500	0.00250	0.000790
1,2-Dibromo-3-chloropropane	0.00530	Charleston Human Health Residential Screening Solid	0.00500	0.00500	0.00250	0.00150
1,2-Dibromoethane (EDB)	0.0360	Charleston Human Health Residential Screening Solid	0.0120	0.00500	0.00250	0.00120
1,2-Dichlorobenzene	0.920	Charleston Eco Screening Solid	0.307	0.00500	0.00250	0.000780
1,2-Dichloroethane	0.460	Charleston Human Health Residential Screening Solid	0.153	0.00500	0.00250	0.00100
1,2-Dichloropropane	1.60	Charleston Human Health Residential Screening Solid	0.533	0.00500	0.00250	0.00140
1,3-Dichlorobenzene	0.740	Charleston Eco Screening Solid	0.247	0.00500	0.00250	0.000620
1,4-Dichlorobenzene	0.890	Charleston Eco Screening Solid	0.297	0.00500	0.00250	0.000440
2-Butanone (MEK)	350	Charleston Eco Screening Solid	117	0.0200	0.0100	0.00590
2-Hexanone	0.360	Charleston Eco Screening Solid	0.120	0.0200	0.0100	0.00480
4-Methyl-2-pentanone (MIBK)	9.70	Charleston Eco Screening Solid	3.23	0.0200	0.0100	0.00590
Acetone	1.20	Charleston Eco Screening Solid	0.400	0.0200	0.0100	0.00510

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Benzene	0.510	Charleston Petroleum Screening Solid	0.170	0.00500	0.00250	0.000920
Bromochloromethane	15.0	Charleston Human Health Residential Screening Solid	5.00	0.00500	0.00250	0.000910
Bromodichloromethane	0.290	Charleston Human Health Residential Screening Solid	0.0967	0.00500	0.00250	0.000600
Bromoform	15.9	Charleston Eco Screening Solid	5.30	0.00500	0.00250	0.000700
Bromomethane	0.235	Charleston Eco Screening Solid	0.0783	0.0100	0.00500	0.00110
Carbon disulfide	0.810	Charleston Eco Screening Solid	0.270	0.00500	0.00250	0.000780
Carbon tetrachloride	0.650	Charleston Human Health Residential Screening Solid	0.217	0.00500	0.00250	0.00130
Chlorobenzene	2.40	Charleston Eco Screening Solid	0.800	0.00500	0.00250	0.000510
Chloroethane	1400	Charleston Human Health Residential Screening Solid	467	0.0100	0.00500	0.00130
Chloroform	0.320	Charleston Human Health Residential Screening Solid	0.107	0.00500	0.00250	0.000350
Chloromethane	10.4	Charleston Eco Screening Solid	3.47	0.0100	0.00500	0.00140
cis-1,2-Dichloroethene	0.0400	Charleston Eco Screening Solid	0.0133	0.00500	0.00250	0.000910
cis-1,3-Dichloropropene	0.398	Charleston Eco Screening Solid	0.133	0.00500	0.00250	0.000720
Cyclohexane	650	Charleston Human Health Residential Screening Solid	217	0.00500	0.00250	0.00140
Dibromochloromethane	2.05	Charleston Eco Screening Solid	0.683	0.00500	0.00250	0.00100
Dichlorodifluoromethane	8.70	Charleston Human Health Residential Screening Solid	2.90	0.0100	0.00500	0.000920
Ethylbenzene	0.810	Charleston Petroleum Screening Solid	0.270	0.00500	0.00250	0.000650
Isopropylbenzene (Cumene)	0.0400	Charleston Eco Screening Solid	0.0133	0.00500	0.00250	0.000920
m,p-Xylene	26.0	Charleston Petroleum Screening Solid	8.67	0.0100	0.00500	0.00170
Methyl acetate	7800	Charleston Human Health Residential Screening Solid	2600	0.00500	0.00300	0.00270

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Methyl tert-butyl ether (MTBE)	0.190	Charleston Petroleum Screening Solid	0.0633	0.00500	0.00250	0.00110
Methylcyclohexane	N/A		0.00500	0.00500	0.00250	0.000960
Methylene chloride	2.60	Charleston Eco Screening Solid	0.867	0.0200	0.0100	0.00790
Naphthalene	1.70	Charleston Petroleum Screening Solid	0.567	0.00500	0.00250	0.000880
o-Xylene	26.0	Charleston Petroleum Screening Solid	8.67	0.00500	0.00250	0.00130
Styrene	1.20	Charleston Eco Screening Solid	0.400	0.00500	0.00250	0.000510
Tetrachloroethene (PCE)	0.180	Charleston Eco Screening Solid	0.0600	0.00500	0.00250	0.00120
Toluene	8.10	Charleston Petroleum Screening Solid	2.70	0.00500	0.00250	0.00140
trans-1,2-Dichloroethene	0.784	Charleston Eco Screening Solid	0.261	0.00500	0.00250	0.000710
trans-1,3-Dichloropropene	0.398	Charleston Eco Screening Solid	0.133	0.00500	0.00250	0.000860
Trichloroethene (TCE)	0.410	Charleston Human Health Residential Screening Solid	0.137	0.00500	0.00250	0.000590
Trichlorofluoromethane	52.0	Charleston Eco Screening Solid	17.3	0.0100	0.00500	0.000910
Vinyl chloride	0.0590	Charleston Human Health Residential Screening Solid	0.0197	0.0100	0.00500	0.000870

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligram per kilogram
N/A = Not Applicable
VOC = Volatile Organic Compound

Matrix: Solid
Analytical Method: pH SW9045D
Concentration level (if applicable): N/A Units: pH Units

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
pH	NA		0.00	0.00	0.00	0.00

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
N/A = Not Applicable

QAPP Worksheet #15b: Eurofins TestAmerica Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Solid
Analytical Method: Gross Alpha & Gross Beta SW9310
Concentration level (if applicable): N/A Units: pCi/g

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Gross Alpha	N/A		10.0	10.0	0.00	0.00
Gross Beta	N/A		10.0	10.0	0.00	0.00

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
N/A = Not Applicable
pCi/g = Picocurie per gram

Matrix: Solid
Analytical Method: Radionuclides GA-01-R
Concentration level (if applicable): N/A Units: pCi/g

Analyte	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Limit of Quantitation (LOQ)	Limit of Detection (LOD)	Method Detection Limit (MDL)
Radium-226	0.00182	Charleston Residential Total PRG	1.00	1.00	0.00	0.00
Radium-228	0.00174	Charleston Residential Total PRG	1.00	1.00	0.00	0.00
Thorium-232	0.00174	Charleston Residential Total PRG	1.00	1.00	0.00	0.00
Thorium-234	0.00178	Charleston Residential Total PRG	2.00	2.00	0.00	0.00
Uranium-235	0.00623	Charleston Residential Total PRG	4.00	4.00	0.00	0.00

Notes:
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
pCi/g = Picocurie per gram

QAPP Worksheet #17: Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

(EPA 2106-G-05 Section 2.3.1)

The primary objective of this phase of the RI will involve sampling of soil and overburden groundwater to determine the presence/absence of contamination at potential source areas. Another objective will be to determine the presence/absence of contamination in the Mountain View Correctional Facility and residential water supply wells in the area. During this phase another objective will be to determine on-Site background concentrations of SVOCs and metals in soil to allow comparison to soils data collected in impacted areas of the Site. In accordance with the DQOs established in **Worksheet #11**, sample locations will target areas with the potential for the presence of contamination.

The data collected from these sampling activities will be used to evaluate the nature and presence/absence of contaminants, update the Conceptual Site Model (CSM), and provide data for screening level human health and ecological risk assessments.

A listing of proposed sample locations for each media, field sample IDs, and rationale for selection of the sampling location is provided on **Worksheet #14/16** and **Worksheet #18**.

QAPP Worksheet #18: Sampling Locations and Methods

(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
01 - Background Soils						
CAFS-01-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-SL004_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-SL005_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-SL006_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL007_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL007_1R2020-DUP	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL007_1R2020-MS	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL007_1R2020-MSD	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL008_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL009_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL010_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL011_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-01-SL012_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL013_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL014_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL015_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL016_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL016_1R2020-DUP	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL016_1R2020-MS	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL016_1R2020-MSD	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL017_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL018_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL019_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-SL020_1R2020	SL	0-0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH001_1R2020	PH	>0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-PH002_1R2020	PH	>0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-PH003_1R2020	PH	>0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-PH004_1R2020	PH	>0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	
CAFS-01-PH005_1R2020	PH	>0.5	Direct Push	Select SVOCs, Metals, Grain size, FOC and pH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-01-PH006_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH007_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH008_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH009_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH010_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH011_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH012_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH013_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH014_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH015_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH016_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH017_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH018_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH019_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH020_1R2020	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH020_1R2020-DUP	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-01-PH020_1R2020-MS	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-01-PH020_1R2020-MSD	PH	>0.5	Direct Push	Select SVOCs and Metals	No. S-11	
CAFS-EB-1_1R2020	EB	NA	NA	Select SVOCs and Metals	No. S-3	
02 - Hill Top Transformer Pads 1 through 10						
CAFS-02-SL001_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL002_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL003_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL004_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL005_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL006_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL007_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL008_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL009_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL010_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-02-SL011_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL011_1R2020-DUP	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL011_1R2020-MS	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL011_1R2020-MSD	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL015_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-02-SL016_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL017_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL018_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL019_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL020_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL021_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL022_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL023_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL024_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL025_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL026_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL027_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL028_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL029_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL030_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL031_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL032_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-02-SL033_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL034_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL035_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL036_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL037_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL038_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL039_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-SL040_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-02-CC001_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC002_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC003_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC004_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC005_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC006_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC007_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC008_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC009_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-02-CC010_1R2020	CC	NA	Grab	PCBs	No. S-19	
CAFS-EB-2_1R2020	EB	NA	NA	PCBs and PCB Congeners	No. S-3	
CAFS-EB-3_1R2020	EB	NA	NA	PCBs	No. S-3	
03 - Hill Top Transformer Poles 1 through 4						

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-03-SL001_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-03-SL002_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-03-SL003_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-03-SL004_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
04 - Hill Top 275 Gallon Waste Oil Tank						
CAFS-04-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC and pH	No. S-11	
CAFS-04-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC and pH	No. S-11	
CAFS-04-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-04-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-04-PH002_1R2020-DUP	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-04-PH002_1R2020-MS	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-04-PH002_1R2020-MSD	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-04-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-04-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-04-PH005_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-04-PH006_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-04-PH007_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-04-PH008_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-04-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-TB-1_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-2_1R2020	TB	NA	NA	VOCs		
05 - Hill Top Former 8,500/10,000 Gallon Fuel Oil UST						
CAFS-05-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH002_1R2020-DUP	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH002_1R2020-MS	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-05-PH002_1R2020-MSD	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-05-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, and pH	No. S-11	
CAFS-05-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-05-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-05-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-05-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-05-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-05-RC001_1R2020	RC	>0.5	Direct Push	VOCs and PCBs	No. S-18	
CAFS-05-RC002_1R2020	RC	>0.5	Direct Push	VOCs and PCBs	No. S-18	
CAFS-05-RC003_1R2020	RC	>0.5	Direct Push	VOCs and PCBs	No. S-18	
CAFS-05-GW001_1R2020	GW	>0.5	Direct Push	VOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-05-GW002_1R2020	GW	>0.5	Direct Push	VOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-05-GW002_1R2020-DUP	GW	>0.5	Direct Push	VOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-05-GW002_1R2020-MS	GW	>0.5	Direct Push	VOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-05-GW002_1R2020-MSD	GW	>0.5	Direct Push	VOCs, PCBs, EPH, and VPH	No. S-11	
CAFS-TB-3_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-4_1R2020	TB	NA	NA	VOCs and VPH		
06 - Hill Top Approximate Location of PCB Contaminated Oil (Water Line Repair 1989)						
CAFS-06-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-06-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-06-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-06-PH003_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-06-PH003_1R2020-DUP	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-06-PH003_1R2020-MS	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-06-PH003_1R2020-MSD	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-06-PH004_1R2020	PH	>0.5	Direct Push	VOCs and PCBs	No. S-11	
CAFS-06-GW001_1R2020	GW	>0.5	Direct Push	VOCs, PCBs, EPH and VPH	No. S-11	
CAFS-TB-5_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-6_1R2020	TB	NA	NA	VOCs and VPH		
07 - Hill Top Two 40,000 Gallon Fuel Oil UST						
CAFS-07-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-07-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-07-PH002_1R2020-DUP	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-07-PH002_1R2020-MS	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-07-PH002_1R2020-MSD	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-07-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-07-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-07-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-07-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-07-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-07-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-07-PH009_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH010_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH011_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH012_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH013_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH014_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH015_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-PH016_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-GW002_1R2020-DUP	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-GW002_1R2020-MS	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-07-GW002_1R2020-MSD	GW	>0.5	Direct Push	VOCs	No. S-11	
08 - Hill Top Lube Oil UST						
CAFS-08-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-08-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size, FOC and pH	No. S-11	
CAFS-08-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-08-PH003_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-08-PH004_1R2020	PH	>0.5	Direct Push	VOCs	No. S-11	
CAFS-08-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
09 - Hill Top Former Radar Buildings						
CAFS-09-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, and pH	No. S-11	
CAFS-09-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, and pH	No. S-11	
CAFS-09-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-09-SL004_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-09-SL005_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-09-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain Size, and pH	No. S-11	
CAFS-09-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain Size, and pH	No. S-11	
CAFS-09-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, and Grain Size	No. S-11	
CAFS-09-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, and Grain Size	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-09-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, and Grain Size	No. S-11	
CAFS-09-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-09-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-09-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-09-GW004_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-09-GW005_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-TB-7_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-8_1R2020	TB	NA	NA	VOCs		
10 - Hill Top Septic Systems Buildings 204, 211, 212 and 213						
CAFS-10-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-10-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-10-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-10-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-10-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-10-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-10-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-10-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-10-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-10-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain size and FOC	No. S-11	
CAFS-10-PH011_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH012_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH012_1R2020-DUP	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH012_1R2020-MS	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH012_1R2020-MSD	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH013_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH014_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH015_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-PH016_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-10-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-10-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-10-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-10-GW004_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-10-TK001_1R2020	TK	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-14	
CAFS-10-TK002_1R2020	TK	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-14	
CAFS-10-TK003_1R2020	TK	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-14	
CAFS-10-TK004_1R2020	TK	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-14	
11 - Hill Top Possible Discharge Pipes Buildings 212 and 213						
CAFS-11-SL001_1R2020	SL	0-0.5	Hand Auger	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-11-SL001_1R2020-DUP	SL	0-0.5	Hand Auger	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-11-SL001_1R2020-MS	SL	0-0.5	Hand Auger	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-1	
CAFS-11-SL001_1R2020-MSD	SL	0-0.5	Hand Auger	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-1	
CAFS-11-SL002_1R2020	SL	0-0.5	Hand Auger	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-1	
CAFS-EB-4_1R2020	EB	NA	NA	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-3	
12 - Hill Top Drum Locations 1 through 5						
CAFS-12-SL001_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-12-SL002_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-12-SL003_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-12-SL004_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-12-SL005_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-1	
CAFS-12-SL006_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL007_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL007_1R2020-DUP	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL007_1R2020-MS	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL007_1R2020-MSD	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL008_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL009_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-12-SL010_1R2020	SL	0-0.5	Hand Auger	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-1	
CAFS-EB-5_1R2020	EB	NA	NA	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-3	
CAFS-TB-9_1R2020	TB	NA	NA	VOCs and VPH		
13 - Small Arms Range						
CAFS-13-SL001_1R2020	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	
CAFS-13-SL001_1R2020-DUP	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	
CAFS-13-SL002_1R2020	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	
CAFS-13-SL003_1R2020	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-13-SL004_1R2020	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	
CAFS-13-SL005_1R2020	SL	0-0.5	Hand Auger	Metals, Grain Size and XRF	No. S-1	
CAFS-13-SL006_1R2020	SL	0-0.5	Hand Auger	Metals and XRF	No. S-1	
CAFS-13-SL007_1R2020	SL	0-0.5	Hand Auger	Metals and XRF	No. S-1	
CAFS-13-SL008_1R2020	SL	0-0.5	Hand Auger	Metals and XRF	No. S-1	
CAFS-13-SL009_1R2020	SL	0-0.5	Hand Auger	Metals and XRF	No. S-1	
CAFS-13-SL010_1R2020	SL	0-0.5	Hand Auger	Metals and XRF	No. S-1	
CAFS-13-SL011_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL011_1R2020-DUP	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL012_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL013_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL014_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL015_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL016_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL017_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL018_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL019_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-13-SL020_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL021_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL022_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL023_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL024_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL025_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL026_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL027_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL028_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL029_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL030_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL031_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL032_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL033_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL034_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL035_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL036_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-13-SL037_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL038_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL039_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL040_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL041_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL042_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL043_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL044_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL045_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL046_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL047_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-13-SL048_1R2020	SL	0-0.5	Hand Auger	XRF	No. S-1	
CAFS-EB-6_1R2020	EB	NA	NA	Metals	No. S-3	
14 - Industrial Area - Fuel Tanks/Coal yard/Coal Yard Runoff Area (2 and 5)						
CAFS-14-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC and pH	No. S-11	
CAFS-14-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-14-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-14-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-14-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-14-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-14-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH011_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-PH012_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-14-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-14-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-14-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
15 - Industrial Area - Water Treatment Plant/Septic Plant/Weld Shop Area (10, 11 and 16)						
CAFS-15-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-15-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-15-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-15-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-15-PH002_1R2020-DUP	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-15-PH002_1R2020-MS	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-15-PH002_1R2020-MSD	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-15-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-15-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-15-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH011_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-PH012_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-15-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-15-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-15-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-TB-10_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-11_1R2020	TB	NA	NA	VOCs		
16 - Industrial Area - Water Treatment Plant/Septic Sand Filter Area (18)						
CAFS-16-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-16-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-16-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH,	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
				Radiological, Grain size, FOC, and pH		
CAFS-16-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-16-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Radiological, Grain size, FOC, and pH	No. S-11	
CAFS-16-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH011_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-PH012_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-16-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-16-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-16-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
17 - Industrial Area - Auto Storage building (19)						
CAFS-17-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-17-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-17-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-17-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-17-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-17-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
18 - Industrial Area - Auto maintenance Shop/Motor pool (4) and Former 2,000 Gallon UST						
CAFS-18-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-18-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH, and VPH	No. S-11	
CAFS-18-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-18-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-18-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH, VPH, Grain size, FOC, and pH	No. S-11	
CAFS-18-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-18-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH009_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH010_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH011_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-PH012_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-18-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-18-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-18-GW003_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-EB-7_1R2020	EB	NA	NA	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-3	
CAFS-TB-12_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-13_1R2020	TB	NA	NA	VOCs		
19 - Industrial Area - Fire Station Building (22)						
CAFS-19-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-19-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-19-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-19-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-19-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-19-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
20 - Industrial Area - Maintenance Shop (15)					No. S-11	
CAFS-20-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-20-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-20-SL002_1R2020-DUP	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-20-SL002_1R2020-MS	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-20-SL002_1R2020-MSD	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, EPH and VPH	No. S-11	
CAFS-20-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain Size, and FOC	No. S-11	
CAFS-20-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, VPH, Grain Size, and FOC	No. S-11	
CAFS-20-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-20-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-20-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-20-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-20-PH007_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-20-PH008_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-20-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-20-GW002_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
21 - Industrial Area - Location of 2,000 Gallon Tank, Use Unknown						
CAFS-21-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-21-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, PCB Congeners, EPH and VPH	No. S-11	
CAFS-21-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-21-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-21-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, PCBs, EPH, and VPH	No. S-11	
CAFS-21-GW001_1R2020	GW	>0.5	Direct Push	VOCs	No. S-11	
CAFS-TB-14_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-15_1R2020	TB	NA	NA	VOCs		
22 - Industrial Area Transformer Poles 5 and 6						
CAFS-22-SL001_1R2020	SL	0-0.5	Hand Auger	PCBs and PCB Congeners	No. S-1	
CAFS-22-SL002_1R2020	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-22-SL002_1R2020-DUP	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-22-SL002_1R2020-MS	SL	0-0.5	Hand Auger	PCBs	No. S-1	
CAFS-22-SL002_1R2020-MSD	SL	0-0.5	Hand Auger	PCBs	No. S-1	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
23 - Former Radio Receiver Building						
CAFS-23-SL001_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, VPH, Grain Size and FOC	No. S-11	
CAFS-23-SL002_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, VPH, Grain Size and FOC	No. S-11	
CAFS-23-SL003_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-23-SL004_1R2020	SL	0-0.5	Direct Push	Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-23-PH001_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, VPH, Grain Size and FOC	No. S-11	
CAFS-23-PH002_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, VPH, Grain Size and FOC	No. S-11	
CAFS-23-PH003_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, VPH, Grain Size and FOC	No. S-11	
CAFS-23-PH004_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-23-PH005_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-23-PH006_1R2020	PH	>0.5	Direct Push	VOCs, Select SVOCs, Metals, EPH, and VPH	No. S-11	
CAFS-23-PH007_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH007_1R2020-DUP	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH007_1R2020-MS	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH007_1R2020-MSD	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH008_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH009_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH010_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-23-PH011_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-23-PH012_1R2020	PH	>0.5	Direct Push	VOCs, EPH, and VPH	No. S-11	
CAFS-TB-16_1R2020	TB	NA	NA	VOCs and VPH		
24 through 27 - Seep Sampling (16 Seep Locations, 4 Rounds)						
CAFS-24-SE001_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE002_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE002_1R2020-DUP	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE002_1R2020-MS	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE002_1R2020-MSD	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE003_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE004_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE005_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE006_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE007_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-24-SE008_1R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE001_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE002_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE003_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-25-SE004_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE004_2R2020-DUP	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE004_2R2020-MS	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE004_2R2020-MSD	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE005_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE006_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE007_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-25-SE008_2R2020	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE001_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE002_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE003_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE004_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE005_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE006_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE007_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-26-SE008_3R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE001_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-27-SE002_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE003_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE004_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE005_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE006_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE007_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-27-SE008_4R2021	SE	GS	Seep Sampling	VOCs, EPH, and VPH	No. S-14	
CAFS-TB-17_1R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-18_2R2020	TB	NA	NA	VOCs and VPH		
CAFS-TB-19_3R2021	TB	NA	NA	VOCs and VPH		
CAFS-TB-20_4R2021	TB	NA	NA	VOCs and VPH		
28 - Correctional Facility Supply Wells and Old Restaurant Well (4 Wells, 4 Rounds)						
CAFS-28-PW001_1R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_1R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_1R2020-DUP	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_1R2020-MS	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_1R2020-MSD	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW003_1R2020	PW	NA	Private Well	VOCs	No. S-16	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-28-PW004_1R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW001_2R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_2R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW003_2R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW004_2R2020	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW001_3R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_3R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW003_3R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW004_3R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW001_4R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW002_4R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW003_4R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-28-PW004_4R2021	PW	NA	Private Well	VOCs	No. S-16	
CAFS-TB-21_1R2020	TB	NA	NA	VOCs		
CAFS-TB-22_2R2020	TB	NA	NA	VOCs		
CAFS-TB-23_3R2021	TB	NA	NA	VOCs		
CAFS-TB-24_4R2021	TB	NA	NA	VOCs		
29 - Residential Wells (6 Wells - 4 Rounds)						
CAFS-29-RE001_1R2020	RE	NA	Private Well	VOCs	No. S-16	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-29-RE002_1R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE003_1R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE004_1R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE005_1R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE006_1R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE001_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_2R2020-DUP	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_2R2020-MS	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_2R2020-MSD	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE003_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE004_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE005_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE006_2R2020	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE001_3R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_3R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE003_3R2021	RE	NA	Private Well	VOCs	No. S-16	

Sample ID	Matrix	Depth (ft BGS)	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CAFS-29-RE004_3R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE005_3R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE006_3R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE001_4R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE002_4R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE003_4R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE004_4R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE005_4R2021	RE	NA	Private Well	VOCs	No. S-16	
CAFS-29-RE006_4R2021	RE	NA	Private Well	VOCs	No. S-16	

Notes:

Concrete Chip	CC
Groundwater Grab	GW
Direct Push	PH
Private Well	PW
Rock Chip	RC
Residence	RE
Trip Blank	TB
Equipment Blank	EB
Surface Location	SL
Tank	TK
Seep	SE

QAPP Worksheet #19 & 30a: Katahdin Analytical Services Sample Containers, Preservation, and Hold Times

(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)

Laboratory: Katahdin Analytical Services, 600 Technology Way, Scarborough Maine, Ms. Heather Manz, hmanz@Katahdinlab.com, (207) 874-2400

List any required accreditations/certifications: DoD ELAP; ISO 17025; NELAP (NH ELAP)

Sample Delivery Method: ERPIMS EDDs, analytical data packages

Analyte/ Analyte Group	Matrix	Method/ SOP [1]	Accreditation Expiration Date	Container(s) (number, size & type per sample)[2, 3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
EPH	Aqueous	MA DEP EPH / CA-511, CA-322	02/01/2022	One 1 L Amber glass bottle	HCl to pH <2; Cool to 4 ± 2° C	14 days	40 days	10 business days
	Solid	MA DEP EPH / CA-511, CA-322	02/01/2022	4-oz wide-mouth jar	Cool to ≤ 6 ° C	14 days	40 days	10 business days
VPH	Aqueous	MA DEP VPH / CA-312	02/01/2022	Two 40-ml VOA vial	HCl to pH < 2, Cool to ≤ 6 °C	None	14 days	10 business days
	Solid	MA DEP VPH / CA-312	02/01/2022	Two 40-ml VOA vial	Cool to ≤ 6 ° C	None	28 days	10 business days
PCBs	Aqueous	SW846 3540C, 8082 / CA-524, CA-329	02/01/2022	One 1 L Amber glass bottle	Cool to ≤ 6 °C	1 Year	1 Year	10 business days

Analyte/ Analyte Group	Matrix	Method/ SOP [1]	Accreditation Expiration Date	Container(s) (number, size & type per sample)[2, 3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
PCBs	Solid	SW846 3550C, 8082 / CA-500, CA-329	02/01/2022	4-oz wide-mouth jar [2]	Cool to ≤ 6 °C	1 Year	1 Year	10 business days
SVOCs	Solid	SW846 3550, 8270 SIM / CA-512, CA-213	02/01/2022	4-ounce (oz) wide-mouth jar	Cool to ≤ 6 °C	7 days	40 days	10 business days
pH	Solid	SW846 9045 / CA-709	02/01/2022	2-oz wide-mouth jar [2]	Cool to ≤ 6 °C	NA	24 hours	10 business days
FOC	Solid	SW9060A / CA-736	02/01/2022	2-oz wide-mouth jar	Cool to ≤ 6 °C	NA	28 days	10 business days
Mercury	Solid	SW7471 / CA-611	02/01/2022	2-oz wide-mouth jar	Cool to ≤ 6 °C	None	28 days	10 business days
Metals	Solid	SW846 3050B, 6020A / CA-605, CA-627	02/01/2022	2-oz wide-mouth jar	Cool to ≤ 6 °C	None	180 days	10 business days
VOCs	Aqueous	SW846 8260C / CA-202	02/01/2022	Three 40-ml VOA vials	Cool to ≤ 6 °C	None	14 days	10 business days
	Solid	SW846 5035, 8260C / CA-214, CA-202	02/01/2022	Three 40-ml VOA vials/ One 40-ml VOA vial	Cool to ≤ 6 ° C	None	14 days	10 business days
Grain Size	Soil	ASTM D422 / CA-551	02/01/2022	8-oz wide-mouth jar	Cool to ≤ 6 ° C	None	None	10 business days
TCLP Extraction	Soil	EPA 1311 / CA-209, CA-510	02/01/2022	Share (3) 8 oz. Soil jar / 4 oz. Soil jar (Filled	Cool to ≤ 6 ° C	14 days from collection to TCLP extraction	None	10 business days

Analyte/ Analyte Group	Matrix	Method/ SOP [1]	Accreditation Expiration Date	Container(s) (number, size & type per sample)[2, 3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
TCLP VOCs		EPA 8260B / CA-202		with no-headspace)		None	14 days	10 business days
TCLP SVOCs		EPA 8270D / CA-226				7 days	40 days	10 business days
TCLP Pesticides		EPA 8081B / CA-302				7 days	40 days	10 business days
TCLP Herbicides		EPA 8151 / CA-305				7 days	40 days	10 business days
TCLP Metals		EPA 6010C / CA-608				None	180 days	10 business days
TCLP Mercury		EPA 7470A / CA-615				None	28 days	10 business days
Ignitability	Solid	SW846 1010A / CA-736	02/01/2022	Included in TCLP Bottleware	Cool to ≤ 6 °C	None	14 days	10 business days
Reactive Sulfide	Solid	EPA 9034 / CA-733	02/01/2022	Included in TCLP Bottleware	Cool to ≤ 6 °C	None	7 days	10 business days
Reactive Cyanide	Solid	EPA 9014 / CA-734	02/01/2022	Included in TCLP Bottleware	Cool to ≤ 6 °C	None	14 days	10 business days

Notes:

[1] SOPs from Katahdin will be forwarded to the government upon request.

[2] Multiple analyses may be performed from the same container as long as preservation requirements are identical and there is sufficient sample volume or mass available.

[3] Size and number of sample containers may change at the discretion of the laboratory. However, Wood approval is needed to implement a change to the container size.

°C = Degrees Celsius
EPH = Extractable Petroleum Hydrocarbon
FOC = Fraction Organic Carbon
HCl = Hydrochloric Acid
L = liter
mL = milliliter

NA = Not Applicable
NELAP = National Environmental Laboratory Accreditation Program
NH ELAP = New Hampshire Environmental Laboratory Accreditation Program
oz = Ounce
PCBs = Polychlorinated Biphenyls
QAPP = Quality Assurance Project Plan
SIM = Selected ion monitoring
SM = Standard Methods
SVOC = Semi volatile Organic Compound
TCLP = Toxicity Characteristic Leachate Procedure
VOA = Volatile Organic Analyte
VOC = Volatile Organic Compound
VPH = Volatile Petroleum Hydrocarbons

QAPP Worksheet #19 & 30b: Eurofins TestAmerica Sample Containers, Preservation, and Hold Times

(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)

Laboratory: Eurofins TestAmerica – Subcontract to Katahdin Analytical Services

TestAmerica St. Louis, 13715 Rider Trail North, Earth City, MO 63045, Chenise Lambert Sykes, chenise.lambert-sykes@testamericainc.com, 314-787-8271

List any required accreditations/certifications: DOD ELAP certification

Sample Delivery Method: ERPIMS EDDs, analytical data packages

Analyte/ Analyte Group	Matrix	Method/ SOP [1]	Accreditation Expiration Date	Container(s) (number, size & type per sample)[2, 3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Gross Alpha/Beta	Soil	SW846 9310 / ST-RD-0403	04/06/2022	1x4oz plastic	None	None	None	10 Business Days
Gamma Spec	Soil	HASL 300 GA-01-R / ST-RD-0102	04/06/2022	1x16oz Plastic or Medium Zip Lock	None	None	None	10 Business Days

Notes:
[1] SOPs from Eurofins TestAmerica will be forwarded to the government upon request.
[2] Multiple analyses maybe performed from the same container as long as preservation requirements are identical and there is sufficient sample volume or mass available.
[3] Size and number of sample containers may change at the discretion of the laboratory. However, Wood approval is needed to implement a change to the container size.
oz = Ounce

QAPP Worksheet #20: Field QC Summary

(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses
Soil	VOCs	160	8	8	8	0	1	9	0	194
Soil	SVOCs	207	11	11	11	0	4	0	0	244
Soil	PCBs	212	11	11	11	0	5	0	0	250
Soil	PCB Congeners	73	4	4	4	0	4	0	0	89
Soil	Metals	215	11	11	11	0	5	0	0	253
Soil	Metals (XRF)	48	2	0	0	0	0	0	0	50
Soil	VPH	173	9	9	9	0	3	9	0	212
Soil	EPH	173	9	9	9	0	3	0	0	203
Soil	Radionuclides Gross Alpha/Beta	6	0	0	0	0	0	0	0	6
Soil	Radionuclides Gross Gamma Spec	6	0	0	0	0	0	0	0	6
Soil	pH	54	4	0	0	0	0	0	0	58
Soil	Grain Size	74	4	0	0	0	0	0	0	78
Soil	FOC	59	3	0	0	0	0	0	0	62
Water	VOCs	105	6	6	6	0	0	15	0	138

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses
Water	VPH	35	3	3	3	0	0	6	0	50
Water	EPH	35	3	3	3	0	0	0	0	50
Water	PCBs	3	1	1	1	0	0	0	0	6

QAPP Worksheet #21: Field SOPs

(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

SOP # or reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified for Project? Y/N	Comments
No. S-1	Surface Soil Sampling	Wood	Shovel, Trowel, Hand Augers	N	None
No. S-2	Calibration of Field Instruments for Water Quality Parameters	Wood	Various Calibration Standard Solutions	N	None
No. S-3	Decontamination of Field Equipment	Wood	LiquiNox®, deionized water, scrub brushes, wash basins, aluminum foil, polyethylene sheeting	N	None
No. S-4	Sampling Packing and Shipment	Wood	Coolers, plastic bags, packing tape, strapping tape, bubble wrap, ice, chains of custody	N	None
No. S-5	Sample Chain of Custody Procedure	Wood	Chains of custody, custody seals, sample labels	N	None
No. S-6	Use of Field Logbooks	Wood	Field Logbooks	N	None
No. S-7	Procedure for Description and Identification of Soils	Wood	Soil Classification/Munsell Chart	N	None
No. S-8	Field Preservation of VOA and VPH Soil Samples	Wood	Sample Containers, P&T Grade Methanol, Plastic Syringes	N	None
No. S-9	Soil Headspace Screening Procedure	Wood	Handheld Photoionization Detector, Jars, Bags	N	None
No. S-10	Spilt Spoon/Split Barrel Subsurface Soil Sample Collection and Standard Penetration Test Procedure	Wood	Various drill rigs, split spoon samplers and rods, engineers ruler, engineers tape measure, camera	N	None

SOP # or reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified for Project? Y/N	Comments
No. S-11	Geoprobe® Direct Push Sampling	Wood	Geoprobe® Machines and Tooling	N	None
No. S-12	Calibration Procedure for PID	Wood	PID, SPAN gas, Zero Air	N	None
No. S-13	Borehole Abandonment	Wood	Tremie pipe, grout pump, various cut off tools	N	None
No. S-14	Surface Water Sampling	Wood	Direct Method, Peristaltic Pump, Scoop, Bucket, Dipper	N	None
No. S-15	XRF Analysis	Wood	XRF, Oven, Sieve, Hood	N	None
No. S-16	Private Drinking Water Well Sampling	Wood	Sample Containers, Decontamination Materials	N	None
No. S-17	Drilling and Heavy Equipment Decontamination	Wood	Portable Steam Cleaner, Brushes, Manual Sprayer, Generator	N	None
No. S-18	Methanol Extracted Rock Chip Sampling	Wood	Drilling equipment, rock hammer, chisels, rock saw	N	None
No. S-19	Concrete Chip Sampling	Wood	Hammer drill, masonry hammer, lump hammer, cold chisels	N	None

Notes:
SOP = standard operating procedure
PID = photoionization detector
P&T = purge and trap
SPAN gas = reference gas
XRF = X-ray fluorescence

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
Water Quality Meter (dissolved oxygen [DO], temperature, pH, oxidation reduction potential [ORP], and specific conductivity meter)	Calibration, Testing, and Inspection	SOP No. S-2, Manufacturer's User Guide	Field Operations Lead	Beginning of each sampling day prior to sample collection and at the end of each day after sample collection has been completed.	Most units: Verification of calibration passes if result is within ±20% certified/ expected value	Troubleshoot problem(s), repeat calibration. If check fails again, obtain new unit and calibrate new unit for use. Document in field logbook.
XRF	Calibration, Testing, inspection	SOP No. S-15, XRF Analysis	Authorized User	Daily test before use	Energy Cal pass/fail Instrument Blank Non-detect Continuing Calibration +/- 20% difference	Energy Cal replace if fail Instrument Blank clean/replace Kapton® window Cont. Cal. Adjust calibration factors per element
Water Level Meter	Measure well depth to water table	Manufacturer's User Guide	Field Operations Lead	Daily test before use	Functions using test water	Replace

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
Turbidity Meter	Calibration, Testing, and Inspection	SOP No. S-2, Manufacturer's User Guide	Field Operations Lead	Beginning of each sampling day prior to sample collection and at the end of each day after sample collection has been completed.	Most units: Verification of calibration passes if result is within +20% certified/ expected value	Troubleshoot problem(s), repeat calibration. If check fails again, obtain new unit and calibrate new unit for use. Document in field logbook.
Photoionization Detector	Calibration, Testing, and Inspection	SOP No. S-12 and Manufacturer's User Guide	Field Operations Lead	Daily test before use	Within 5 ppm of zero and within +/- 10% of span gas standard	Troubleshoot problem(s), repeat calibration. If check fails again, obtain new unit and calibrate new unit for use. Document in field logbook.

QAPP Worksheet #23a: Katahdin Analytical SOPs

(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	‡Modified for Project? Y/N
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 01/20, Revision 20.	Definitive	Water and Soil / VOCs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	N
CA-209	Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311, 07/19, Revision 8.	Definitive	Water and Soil / VOCs	Rotary Extractor	N
CA-213	Analysis of Semi volatile Organic Compounds By: SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM), 01/19, Revision 15.	Definitive	Water and Soil / SVOCs and PAHs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	N
CA-214	Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples Using SW846 Method 5035, 02/20, Revision 8.	Definitive	Soil / VOCs	Not applicable (extraction)	N
CA-226	Analysis of SVOAs by Capillary Column GC/MS: SW-846 Method 8270D, 06/17, Revision 10. (Reviewed 01/19)	Definitive	Water and Soil / SVOCs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	N
CA-302	Analysis of Pesticides by Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8081, 10/19, Revision 20	Definitive	Water and Soil / Pesticides	Gas Chromatography (GC)/ Electron Capture Detector (ECD)	N

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	‡Modified for Project? Y/N
CA-305	Analysis Of Chlorinated Herbicides By GC Using Methylation Derivatization: SW-846 Method 8151, 2/20, Revision 15.	Definitive	Water and Soil / Herbicides	Gas Chromatography (GC)/ Electron Capture Detector (ECD)	N
CA-312	Method for the Determination of Volatile Petroleum Hydrocarbons (MADEP - VPH), 09/17, Revision 11. (reviewed 02/19)	Definitive	Water and Soil / VPH	Gas Chromatography (GC)/ Flame Ionization Detector (FID)/ Photo Ionization Detector (PID)	N
CA-322	Method for the Analysis of Extractable Petroleum Hydrocarbons by MADEP – EPH, 10/18, Revision 15. (Reviewed 01/20)	Definitive	Water and Soil / EPH	Gas Chromatography (GC)/ Flame Ionization Detector (FID)	N
CA-329	Analysis Of PCBs As Total Aroclors By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082, 01/19, Revision 19.	Definitive	Water and Soil / PCBs	Gas Chromatography (GC)/ Electron Capture Detector (ECD)	N
CA-500	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Pesticides/PCBs Analysis, 03/19, Revision 11. (Reviewed 01/20)	Definitive	Soil / Pesticides and PCBs	Not applicable (extraction)	N
CA-502	Preparation Of Aqueous Samples For Extractable Semi volatile Analysis, 03/19, Revision 12. (Reviewed 01/20)	Definitive	Water / SVOCs and PAHs	Not applicable (extraction)	N
CA-510	Toxicity Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes, 07/19, Revision 11.	Definitive	Water and Soil / Various	Rotary Extractor	N
CA-511	Extraction of Petroleum Hydrocarbons From Samples for Analysis by MADEP – EPH Methods, 07/19, Revision 13.	Definitive	Water and Soil/EPH	Not applicable (extraction)	N
CA-512	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable	Definitive	Soil / SVOCs and PAHs	Not applicable (extraction)	N

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	‡Modified for Project? Y/N
	Semi-Volatiles Analysis, 04/19, Revision 14.				
CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 01/19, Revision 14.	Definitive	Water / PCBs	Not applicable (extraction)	N
CA-516	Preparation of Aqueous Samples for Herbicides by Method 8151, 03/19, Revision 12.	Definitive	Water / Herbicides	Not applicable (extraction)	N
CA-524	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Pesticide/PCB Analysis, 03/19, Revision 11. (Reviewed 01/20)	Definitive	Soil / Pesticides and PCBs	Not applicable (extraction)	N
CA-526	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semi volatile Analysis, 04/19, Revision 12.	Definitive	Soil / SVOCs and PAHs	Not applicable (extraction)	N
CA-551	Grain Size Analysis, 04/19, Revision 2.	Definitive	Solid / Grain Size	Sieves, Hydrometer	N
CA-604	Acid Digestion of Aqueous Samples by USEPA Method 3010 for ICP Analysis of Total or Dissolved Metals, 01/19, Revision 9.	Definitive	Water / TAL Metals and TAL Metals + Boron	Not applicable (digestion)	N
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 01/19, Revision 8.	Definitive	Soil / TAL Metals	Not applicable (digestion)	N
CA-608	Trace Metals Analysis By ICP-AES Using EPA Method 6010, 01/19, Revision 19.	Definitive	Water and Soil / TAL Metals	Inductively Coupled Plasma (ICP) / Atomic Emission Spectroscopy (AES)	N
CA-611	Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471, 01/19, Revision 12.	Definitive	Soil / Mercury	Mercury Analyzer	N

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	‡Modified for Project? Y/N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470, 01/19, Revision 11.	Definitive	Water / Mercury	Mercury Analyzer	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 01/19, Revision 13.	Definitive	Water and Soil / TAL Metals and TAL Metal	Inductively Coupled Plasma (ICP) / Mass Spectroscopy (MS)	N
CA-709	pH Concentration Measurements In Soil Matrices – SW 846 Method 9045, 10/19, Revision 12.	Definitive	Soil / pH IDW / Corrosivity	pH Meter	N
CA-733	Reactive Cyanide SW-846 Chapter Seven, 7.3.3.2, 05/18, Revision 7. (Reviewed 01/19)	Definitive	IDW / Reactive Cyanide	Konelab	N
CA-734	Reactive Sulfide SW-846 Chapter Seven, 7.3.4.2, 05/12, Revision 7. (Reviewed 01/20)	Definitive	IDW / Reactive Sulfide	Buret	N
CA-736	Test Method for Flash Point by Pensky-Martens Closed-Cup Tester, 07/19, Revision 7. (Reviewed 01/20)	Definitive	IDW / Ignitability	Pensky-Martens Closed-Cup Tester	N
CA-741	Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method, 01/19, Revision 8.	Definitive	Soil / Fractional Organic Carbon	Total Organic Carbon Analyzer	N
SD-902	Sample Receipt and Internal Control, 01/19, Revision 13.	NA	NA	NA	N
SD-903	Sample Disposal, 09/17, Revision 6. (Reviewed 01/20)	NA	NA	NA	N

‡ A brief summary of project-specific SOP modifications must be provided on this worksheet or referenced.

QAPP Worksheet #23b: Eurofins TestAmerica Analytical SOPs

(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

SOP #	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	‡Modified for Project? Y/N
ST-RD-0102	GammaVision Analysis, Revision 18, 03/22/19	Definitive	Soil Gamma Spec	Gamma Spec	N
ST-RD-0403	Low Background Gas Flow Proportional Counting System Analysis, Revision 20, 01/16/19	Definitive	Soil Gross Alpha/Beta	GFPC	N

‡ A brief summary of project-specific SOP modifications must be provided on this worksheet or referenced.

QAPP Worksheet #24a: Katahdin Analytical Services Analytical Instrument Calibration

(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
GC/MS-VOCs	Initial Calibration (ICAL) - A minimum 5-point initial calibration is required for all VOCs.	AQ -1-150 ug/L SL – 5-150 ug/L	Instrument receipt, major instrument change, when continuing calibration verification does not meet criteria.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat calibration.	Analyst, Department Manager.	CA-202
GC/MS-VOCs	Calibration Check Standard (ICV)	NA	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Analyst / Supervisor	CA-202
GC/MS-VOCs	Continuing Calibration (CCV)	NA	Daily before sample analysis and every 12 hours	All reported analytes and surrogates within ± 20% of true value. All	DoD (Department of Defense) project level approval must	Analyst, Department Manager	CA-202

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
				reported analytes and surrogates within ± 50% for end of analytical batch CCV.	be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.		
GC/MS-VOCs	BFB Tune	NA	Prior to ICAL and at the beginning of each 12-hour clock.	Must meet criteria listed in Section 7.3, current revision of SOP CA-202.	Retune and/or clean source.	Analyst, Department Manager	CA-202
GC/MS (full scan) SVOCs	ICAL - A minimum 5-point calibration is required for all SVOCs.	10-125 ug/mL	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria. Six-point initial calibration for all analytes.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte:	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	CA-226

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
				r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.			
GC/MS (full scan) SVOCs	ICV	NA	Once after each ICAL.	The %R must be within 80-120% for all project compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-226
GC/MS (full scan) SVOCs	CCV	NA	Analyze a standard at the beginning of each 12-hour shift after a decafluoro-triphenyl-phosphine (DFTPP) tune.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If	Analyst, Department Manager	CA-226

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
					that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.		
GC/MS (full scan) SVOCs	DFTPP Tune	NA	Every 12 hours	Criteria listed in Section 7.4, current revision of SOP CA-226	Retune and/or clean source.	Analyst, Department Manager	CA-226
GC/ECD-Pesticides	ICAL - A minimum 5-point calibration for all individual pesticides, Toxaphene and Technical Chlordane	0.005-0.25 ug/mL Tox: 0.1-10 ug/mL TC: 0.05-2.5 ug/mL	Instrument receipt, major instrument change, when CCV does not meet criteria.	One of the options below: Option 1: %RSD for each analyte must be ≤ 20%; Option 2: linear least squares regression: r must be ≥ 0.995; Option 3: non-linear regression: r^2 must be ≥ 0.99 (6 points shall be used for second order).	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	CA-302

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
GC/ECD-Pesticides	ICV	NA	Immediately following ICAL.	%R must within 80%-120% for all project compounds.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-302
GC/ECD-Pesticides	CCV	NA	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	%D must be ≤ 20% for all project compounds.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-302
GC/ECD-Pesticides	Breakdown Check	NA	Perform daily prior to sample analysis.	The degradation must be ≤ 15% for both Endrin and DDT.	Column maintenance; injection port maintenance.	Analyst, Department Manager	CA-302
GC/ECD-Herbicides	ICAL – A minimum 5-point calibration of all herbicides	0.1/10-2.0/200 ug/mL	Instrument receipt, major instrument change, when CCV does not meet criteria.	One of the options below: Option 1: %RSD for each analyte must be ≤ 20%; Option 2: linear least squares regression: r must be ≥	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	CA-305

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
				0.995; Option 3: non-linear regression: r^2 must be ≥ 0.99 (6 points shall be used for second order).			
GC/ECD-Herbicides	ICV	NA	Immediately following calibration.	%R must within 80%-120% for all project compounds.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-305
GC/ECD-Herbicides	CCV	NA	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D must be ≤ 20% for all project compounds.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-305
GC/ECD-PCBs	ICAL - A minimum 5-point calibration is run except for mid-point calibration of Aroclors 1221 and 1232; if	0.5-10 ug/mL	Instrument receipt, major instrument change, when CCV does not meet criteria.	One of the options below: Aroclors 1016/1260, 1242, 1248, and 1254 – One of the options below:	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-329

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
	targets are detected, then 6-point calibration is performed.			Option 1: %RSD for each analyte must be ≤ 20%; Option 2: linear least squares regression: r must be ≥ 0.995; Option 3: non-linear regression: r2 must be ≥ 0.99 (6 points shall be used for second order)	Reanalyze affected data.		
GC/ECD-PCBs	ICV	NA	Immediately following ICAL.	%R must within 80%-120% for all project compounds.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-329
GC/ECD-PCBs	CCV	NA	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	%D must be ≤ 20% for all project compounds.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-329

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
GC/ECD-PCBs as congeners	ICAL - A minimum 5-point calibration is required.	0.005-0.25 ug/mL	Instrument receipt, major instrument change, when CV does not meet criteria.	One of the options below: Option 1: %RSD for each analyte must be ≤ 20%; Option 2: linear least squares regression: r must be ≥ 0.995; Option 3: non-linear regression: r^2 must be ≥ 0.99 (6 points shall be used for second order).	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	CA-334
GC/ECD-PCBs as congeners	ICV	NA	Immediately following ICAL.	%R must be within 80-120% for all project analytes.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-334
GC/ECD-PCBs as congeners	CCV	NA	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	%D must be ≤ 20% for all project compounds.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful	Analyst, Department Manager	CA-334

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
					calibration verification.		
ICP-AES – TAL Metals	ICAL	0.1-2.5 mg/L	At the beginning of each day or if QC is out of criteria.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-608
ICP-AES – TAL Metals	ICV	NA	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-608
ICP-AES – TAL Metals	Calibration Blank (CB)	NA	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	CA-608

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
ICP-AES – TAL Metals	CCV	NA	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-608
ICP-AES – TAL Metals	Low-level Calibration Check Standard (if using one-point ICAL)	NA	Daily after one-point ICAL.	%R must within 80%-120% for all project compounds.	Correct problem, then reanalyze.	Analyst, Department Manager	CA-608
ICP-AES – TAL Metals	ICS - ICSA & ICSB	NA	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	CA-608
ICP-MS – TAL Metals	Tune	NA	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value.	Perform necessary equipment maintenance.	Analyst, Department Manager	CA-627

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
				Resolution must be <0.9 amu full width at 10% peak height. Four injections %RSD must be <5%.			
ICP-MS – TAL Metals	ICAL - 1 point calibration plus blank	0.05-10 mg/L	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-627
ICP-MS – TAL Metals	ICV	NA	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% for all project compounds.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-627
ICP-MS – TAL Metals	CCB	NA	Before beginning a sample sequence, after every 10 samples and at end of the analysis	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	CA-627

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
			sequence. For negative blanks, absolute value < LOD.				
ICP-MS – TAL Metals	CCV	NA	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project compounds.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-627
ICP-MS – TAL Metals	ICS - ICSA & ICSB	NA	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICS-AB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	CA-627
Mercury analyzer	ICAL - 5 points plus a calibration blank	0.2-10 ug/L	Upon instrument receipt, major instrument change, at the start of each day.	Correlation coefficient (r) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-611, CA-615

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
Mercury analyzer	ICV	NA	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110%	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-611, CA-615
Mercury analyzer	CCB	NA	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	CA-611, CA-615
Mercury analyzer	CCV	NA	Beginning and end of each run sequence and every 10 samples.	%R must be within 80-120%	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-611, CA-615

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
Probe	3-point calibration with pH buffers with a midrange cal. check	pH 4, 7 & 12	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	Analyst, Department Manager	CA-708. CA-709
Probe	ICV	NA	One per batch of twenty or fewer samples	± 0.1 pH units	Correct problem, recalibrate	Analyst, Department Manager	CA-708. CA-709
Buret / Sulfide	Standardized using 0.0375 N Sodium thiosulfate	NA	Daily prior to sample analysis.	NA	An acceptable titrant is compared against an independent source identified as an LCS	Analyst, Supervisor	CA-722
Konelab – Cyanide	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	10-250 ug/L	Daily ICAL prior to sample analysis.	Correlation coefficient (r) must be ≥ 0.995.	Correct problem, then repeat ICAL.	Analyst, Department Manager	CA-772
Konelab – Cyanide	Distilled Standards (Cyanide only)	10-250 ug/L	One low point and one low point per multipoint calibration.	%R must be within 85-115%.	Correct problem, then repeat distilled standards.	Analyst, Department Manager	CA-772

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
Konelab – Cyanide	ICV	NA	Once after each ICAL, prior to beginning a sample run.	%R must be within 85-115%.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-773
Konelab – Cyanide	CCV (undistilled)	NA	One after every 10 samples analyzed and at close of run	90%-110 %R.	Correct problem and verify CCV. Rerun CCV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-773
VPH	ICAL	1-300 ug/L C9-C10: 1-300 ug/L C5-C8: 3-900UG/l C9-C12: 2-600 UG/l	Instrument receipt, major instrument change, when CCV does not meet criteria.	The %RSD must be ≤ 25%.	Investigate and repeat ICAL.	Analyst, Department Manager	CA-312
VPH	ICV	NA	Once after each initial calibration.	The %D of the expected value must be ≤25%for all analytes.	Reanalyze standard. Reprepare standard. Reprepare standard from fresh stock.	Analyst, Department Manager	CA-312

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
VPH	CCV	NA	Analyze prior to sample analysis, after every 20 samples and at end of sequence.	The %D must be ≤30% for n-nonane and ≤25% for all other analytes.	Evaluate the samples: If the %D >25% (30% for n-nonane) and sample results are < LOQ, narrate. Otherwise, reanalyze all samples after last acceptable CV.	Analyst, Department Manager	CA-312
EPH	ICAL	1-200 ug/mL C9-C18: 6-1200 ug/mL C19-C36: 8-1600 ug/mL C11-C22: 17-3400 ug/mL	Prior to sample analysis.	The %RSD must be ≤ 25% or the r must be ≥ 0.99.	Investigate and repeat ICAL.	Analyst, Department Manager	CA-322
EPH	ICV	NA	Immediately following calibration.	The %D of the expected value must be ≤25%for all analytes.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-322
EPH	CCV	NA	After every 20 samples; If calibration curve previously analyzed, analyze daily before samples.	The %D must be ≤30% for n-nonane and ≤25% for all other analytes. The closing CCV may have four analytes >	Evaluate the samples: If the %D >25% (30% for n-nonane) and sample results are < LOQ, narrate. Otherwise,	Analyst, Department Manager	CA-322

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
				than 25%D but < 40%D.	reanalyze all samples after last acceptable CCV.		
Total Organic Carbon Analyzer / Fraction Organic Carbon	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	100 ug – 24000 ug	Initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient ≤ 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	CA-741
Total Organic Carbon Analyzer / Fraction Organic Carbon	ICV	NA	Once after each ICAL, prior to beginning a sample run.	Lloyd Kahn: %R must within 80%-120% SM5310B: %R must within 90%-110%	(1) If the ICV fails high, report samples that are <PQL. (2) Redigest, recalibrate and/or reanalyze other samples.	Analyst, Department Manager	CA-741
Total Organic Carbon Analyzer / Fraction Organic Carbon	CCV	NA	Every 10 samples and at the end of the run	Lloyd Kahn: %R must within 80%-120% SM5310B: %R must within 90%-110%	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last	Analyst, Department Manager	CA-741

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
					acceptable CCV recovery.		

Notes:
BFB = 4-bromofluorobenzene
CV = Calibration Verification
GC/ECD = Gas Chromatography Electron Capture Detector
EPH = Extractable Petroleum Hydrocarbons
GC = Gas Chromatography
GC-MS = Gas Chromatography– Mass Spectrometer]
ICP-AES = inductively coupled Plasma – Atomic Emission Spectrometer
ICP-MS = inductively coupled Plasma – Mass Spectrometer
ICS = Interference Correction Standard
ICSA = Interference Correction Standard A
ICSAB = Interference Correction Standard AB
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
NA = Not Applicable
PCBs = Polychlorinated Biphenyls
RSD = Relative Standard Deviation
SIM = Selective Ion Monitoring
SOP = Standard Operating Procedure
SVOC = Semi volatile Organic Compound
TAL = Target Analyte List
VOC = Volatile Organic Compound
VPH = Volatile Petroleum Hydrocarbons
%D = Percent Deviation
%R = Percent Recovery

QAPP Worksheet #24b: Eurofins TestAmerica Analytical Instrument Calibration

(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
Gamma Spectrometer	Initial Calibration Verification (ICAL) for Energy, Efficiency, and FWHM peak resolution	Range: the energy range is determined by the sample matrix (e.g. 46.54 to 1836.1 keV). The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration. The curve should have at least eight points to determine the efficiency.	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings	Peak energy difference is within 0.1 keV of reference energy for all points. Peak FWHM < 2.5 keV at 1332 keV. Energy vs channel slope equation shall be linear and accurate to 0.5 keV	Correct problem, then repeat ICAL.	Lab Manager / Analyst	ST-RD-0102

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
		The calibration source must have radionuclides that "bracket" the intended range of calibration. A minimum of 10,000 counts will be accumulated for each data point.					
Gamma Spectrometer	Initial Calibration Verification (ICV)	At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).	After ICAL for energy/efficiency and prior to analysis of samples.	Observed peaks of second source standard fall within ± 10% of initial calibration value relative to the true value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV as appropriate.	Lab Manager / Analyst	ST-RD-0102
Gas Flow Proportional Counter	Initial Calibration - Voltage Plateau (ICALV)	Prior to initial use and after loss of control.	Slope of the plateau less than 5% over a range of 100V.	Correct problem, then repeat ICALV.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
	(separate plateaus determined for alpha and beta activity)						
Gas Flow Proportional Counter	Initial Calibration - Efficiency (ICALE)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using electroplated sources.	Correct problem, then repeat ICALE.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter
Gas Flow Proportional Counter	Initial Calibration – Cross-talk Factors (ICALCT)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	Verify manufacturer's specifications for cross talk in alpha and beta channels.	Correct problem, then repeat ICALCT.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter
Gas Flow Proportional Counter	Initial Calibration – Self-Absorption Curve (ICALSA)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	For each radionuclide of interest (or isotope with similar energy profile), establish mathematical function (curve) of detector efficiency vs. source mass loading. Best fit of	Correct problem, then repeat ICALSA.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
			data with coefficient of determination (r2) closest to 1.00 and the smallest standard error				
Gas Flow Proportional Counter	Efficiency Calibration Verification (IECV)	After ICALE for alpha and beta and prior to analysis of samples.	Value of second source calibration for each isotope within ±10% of initial calibration value.	Correct problem and verify second source standard. Rerun IECV. If that fails, correct problem and repeat ICALE.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter
Gas Flow Proportional Counter	Continuing Calibration Verification (CCV)	After a counting gas change and daily for short test-source counting intervals.	Within tolerance or control chart limits ± 3% or 3σ of the mean.	Correct problem, rerun calibration verification. If that fails, then repeat ICALE. Reanalyze all samples since the last successful calibration verification.	Lab Manager / Analyst	ST-RD-0403	Gas Flow Proportional Counter

QAPP Worksheet #25a: Katahdin Analytical Instrument and Equipment Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
GC/MS VOCs	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-202
GC/MS SVOCs	Check pressure and gas supply daily. Manual	SVOCs	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat	Analyst, Department Manager	CA-226

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
	tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.					calibration or CCV		
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Pesticides and Herbicides	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-302, CA-305, CA-329

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
GC/FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Diesel Range Organics	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-322
GC/PID/FID	Replace or cut GC column as needed. Bake out trap and column. Change trap as needed.	Gasoline Range Organics	Trap, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-312
Sieves	Cleaning	Grain Size	Visual inspection for clogs or tears	Each use	N/A	Remove from service	Analyst, Department Manager	CA-551
TOC Combustion Analyzer	Check level of dilution water, drain vessel	Fractional Organic Carbon	Tubing, sample boat, syringe,	Prior to initial calibration	Acceptable calibration or CCV	Correct the problem and repeat	Analyst, Department Manager	CA-741

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
	water, humidifier water, auto sampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.		humidifier, rinse reservoir, phosphoric acid vessel, oxygen pressure	and as necessary		calibration or CCV		
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-302, CA-305, CA-329

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-608
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed.	TAL Metals	Torch, nebulizer, spray chamber, pump tubing.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-627

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
	Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.							
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-611, CA-615

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
Probe - pH	Clean, drain, and refill reference electrode as needed.	pH	Reference electrode for white crystals, Inspect electrode for damage.	Before use	pH 7 ± 0.05 pH units (pH)	Correct problem and repeat calibration.	Analyst, Department Manager	CA-708, CA-709
Buret – Reactive Sulfide	N/A	Reactive Sulfide	Visual inspection for cracks or chips	Each use	N/A	Remove from service	Analyst, Department Manager	CA-722
Konelab	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, change dispensing needle, all as needed.	Reactive Cyanide	Reagent tubes, lamp, wash tubes, paddles, syringes, dispensing needles.	Prior to initial calibration and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-772
Pensky-Martens	NA	Flashpoint	NA	NA	Acceptable LCS or CCV	Correct the problem and repeat LCS or CCV	Analyst, Department Manager	CA-736

QAPP Worksheet #25b: Eurofins TestAmerica Analytical Instrument and Equipment Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
Gas Flow Proportional Counter	1. Clean instrument	1. Physical check	1. Daily	1. Daily	1. None applicable	Recalibrate	TestAmerica Analyst	ST-RD-0403
Gas Flow Proportional Counter	2. Inspect windows	2. Physical check	2. High counts and/or background	2. High counts and/or background	2. No physical defects	Instrument maintenance	TestAmerica Analyst	ST-RD-0403
Gas Flow Proportional Counter	3. QA check	3. Background and source count	3. Daily	3. Daily	3. Within 3 sigma of 20 day population	Consult with Technical Director	TestAmerica Analyst	ST-RD-0403
Gamma Spectrometer	1. Clean cave; fill dewar with N2	1. Physical check	1. Physical check	1. Weekly	1. Acceptable background	Recalibrate	TestAmerica Analyst	ST-RD-0102
Gamma Spectrometer	2. QA check	2. Background and source check	2. Check deviation	2. Daily	2. Within 3 sigma of measured population	Instrument maintenance and consult with Technical director	TestAmerica Analyst	ST-RD-0102

QA = quality assurance

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal

(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)

Sampling Organization: Wood
Laboratory: Katahdin Analytical Services
Method of sample delivery (shipper/carrier): Katahdin Courier Service
Number of days from reporting until sample disposal: 60 Days from Receipt

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	Wood – Field Operations Lead	NA
Chain-of-custody form completion	Wood – Field Operations Lead	S-8
Packaging	Wood – Field Operations Lead	S-7
Courier coordination	Wood – Field Operations Lead	NA
Courier coordination	Katahdin Analytical Services – PM, Login Supervisor	NA
Sample receipt, inspection, & log-in	Katahdin Analytical Services – PM, Login Supervisor, Sample receipt personnel	SD-902
Sample custody and storage	Katahdin Analytical Services – PM, Login Supervisor, Sample receipt personnel	SD-902
Sample disposal	Katahdin Analytical Services – PM, Login Supervisor, Sample receipt personnel	SD-903

Notes:
PM = project manager
NA = not applicable

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260C/ CA-202**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method Blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260C/ CA-202**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Surrogate	Four per sample: Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260C/ CA-202**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
		limits are not specified.				
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260C/ CA-202**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

Notes:
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
QC = Quality Control
SOP = Standard Operating Procedure
VOC = Volatile Organic Compound

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**SVOCs**
Analytical Method/ SOP Reference	**SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**SVOCs**
Analytical Method/ SOP Reference	**SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Surrogate	Full Scan - 6 per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14 SIM – 4 per sample: 2-Methylnaphthalene-d10 Fluorene-d10 Pyrene-d10 2,4-Dibromophenol	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**SVOCs**
Analytical Method/ SOP Reference	**SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**SVOCs**
Analytical Method/ SOP Reference	**SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
		specified. RPD of all analytes = 20% (between MS and MSD				

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**SVOCs**
Analytical Method/ SOP Reference	**SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
IS	Six per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

Notes:

DL = Detection Limit
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
SOP = Standard Operating Procedure
SVOC = Semi volatile Organic Compound

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Pesticides, Herbicides and PCBs**
Analytical Method/ SOP Reference	**SW846 8081B, 8082A, 8151A / CA-302, CA-305, CA-329**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Pesticides, Herbicides and PCBs**
Analytical Method/ SOP Reference	**SW846 8081B, 8082A, 8151A / CA-302, CA-305, CA-329**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	Pesticides and PCBs - two per sample: Decachloro-biphenyl Tetrachloro-m-xylene Herbicides – one per sample: 2,4-Dichlorophenylacetic acid	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Pesticides, Herbicides and PCBs**
Analytical Method/ SOP Reference	**SW846 8081B, 8082A, 8151A / CA-302, CA-305, CA-329**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
LCS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Pesticides, Herbicides and PCBs**
Analytical Method/ SOP Reference	**SW846 8081B, 8082A, 8151A / CA-302, CA-305, CA-329**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MS/MSD	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%.	None. Apply qualifier if RPD >40% and discuss in the case narrative. The higher of the two results will be reported unless matrix interference is apparent.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Pesticides, Herbicides and PCBs**
Analytical Method/ SOP Reference	**SW846 8081B, 8082A, 8151A / CA-302, CA-305, CA-329**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

Notes:

DL = Detection Limit

IDW = Investigation Derived Waste

LCS = Laboratory Control Sample

LOD = Limit of Detection

LOQ = Limit of Quantitation

PCBs = Polychlorinated Biphenyls

QC = Quality Control

QSM = Quality Systems Manual

RPD = Relative Percent Difference

SOP = Standard Operating Procedure

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater and Soil**
Analytical Group	**VPH**
Analytical Method/ SOP Reference	**MADEP VPH / CA-312**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected > LOQ	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report sample results which are < LOQ and >10X the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater and Soil**
Analytical Group	**VPH**
Analytical Method/ SOP Reference	**MADEP VPH / CA-312**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Surrogates	2,5-Dibromotoluene	Water and soil: 70-130% recovery	Reanalyze; present both sets of data.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS/LCSD	One per preparation batch of twenty or fewer samples of similar matrix.	Water and soil: 70-130% recovery. RPD ≤25	(1) Locate source of problem; re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater and Soil**
Analytical Group	**VPH**
Analytical Method/ SOP Reference	**MADEP VPH / CA-312**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS/LCSD	One per preparation batch of twenty or fewer samples of similar matrix.	Water and soil: 70-130% recovery. RPD ≤25	(2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-analysis is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>130%), re-analysis is not required if affected compounds were not detected in associated samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater and Soil**
Analytical Group	**VPH**
Analytical Method/ SOP Reference	**MADEP VPH / CA-312**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	Water and soil: 70-130% recovery. RPD ≤ 50%	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Analyst, Supervisor, QA Manager	Precision/Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Notes:
DL = Detection Limit
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MADEP = Massachusetts Department of Environmental Protection
QA = Quality Assurance
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference
SOP = Standard Operating Procedure
VPH = Volatile Petroleum Hydrocarbons

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater and Soil**
Analytical Group	**EPH**
Analytical Method/ SOP Reference	**MA DEP EPH / CA-322**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected > LOQ	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report sample results which are < LOQ and >10X the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	ortho-Terphenyl, 5-alpha-androstane, 2-Fluorobipheny 2-bromonaphthalene	Water and soil: 40-140% recovery for all 3 surrogates	Reanalyze; present both sets of data.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

LCS/LCSD	One per preparation batch of twenty or fewer samples of similar matrix.	Water and soil: 40-140% recovery	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples. (4) Re-fractionate archived batch extracts if either the concentration of naphthalene and/or 2-methylnaphthalene in aliphatic fraction is >5% of either of their respective total concentrations.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	Water and soil: 40-140% recovery; RPD ≤ 50%	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Analyst, Supervisor, QA Manager	Precision/Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Notes:

DL = Detection Limit

IDW = Investigation Derived Waste

EPH = Extractable Petroleum Hydrocarbons

LCS = Laboratory Control Sample

LOD = Limit of Detection

LOQ = Limit of Quantitation

MADEP = Massachusetts Department of Environmental Protection

QA = Quality Assurance

QC = Quality Control

QSM = Quality Systems Manual

RPD = Relative Percent Difference

SOP = Standard Operating Procedure

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Matrix Spike	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Post-digestion Spike	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-AES)**
Analytical Method/ SOP Reference	**SW-846 6010C / CA-608**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

Notes:
CA = Corrective Action
DL = Detection Limit
ICP-AES = inductively coupled Plasma – Atomic Emission Spectrometer
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual
RPD = Relative Percent Difference
SOP = Standard Operating Procedure

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
MS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
MSD	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Post-digestion Spike	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution (not applicable for rinsate blanks)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and/or IDW**
Analytical Group	**Metals (ICP-MS)**
Analytical Method/ SOP Reference	**SW846 6020A / CA-627**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30-120% of that of initial calibration standard.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Per the method, for each sample, IS intensity must be ≥ 70% of that of initial calibration standard.

Notes:
QSM = Quality Systems Manual
RPD = Relative Percent Difference
SOP = Standard Operating Procedure
CA = Corrective Action
DL = Detection Limit
ICP-MS = inductively coupled Plasma – Mass Spectrometer
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
QC = Quality Control

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Metals (Mercury)**
Analytical Method/ SOP Reference	**SW-846 7470A/7471B/ CA-611, CA-615**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Metals (Mercury)**
Analytical Method/ SOP Reference	**SW-846 7470A/7471B/ CA-611, CA-615**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS	One per preparatory batch.	AQ: 82-119 %R SL: 80-124	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	Same as LCS RPD of all analytes = 20% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Notes:
AQ = Aqueous
CA = Corrective Action
DL = Detection Limit
ICP-MS = inductively coupled Plasma – Mass Spectrometer
IDW = Investigation Derived Waste
RPD = Relative Percent Difference
SL = Soil
SOP = Standard Operating Procedure
%R = Percent Recovery
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
QC = Quality Control
QSM = Quality Systems Manual

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Soil**
Analytical Group	**Fraction Organic Carbon**
Analytical Method/ SOP Reference	**Lloyd Kahn/ CA-741**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil**
Analytical Group	**Fraction Organic Carbon**
Analytical Method/ SOP Reference	**Lloyd Kahn/ CA-741**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Control Sample	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Matrix Spike	One for every set 10 samples	%R must be within: 75-125	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil**
Analytical Group	**Fraction Organic Carbon**
Analytical Method/ SOP Reference	**Lloyd Kahn/ CA-741**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Quadruplicate	One sample duplicate per 20 samples.	RPD ≤30 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Notes:

CA = Corrective Action

DL = Detection Limit

IDW = Investigation Derived Waste

LCS = Laboratory Control Sample

LOD = Limit of Detection

LOQ = Limit of Quantitation

QC = Quality Control

QSM = Quality Systems Manual

RPD = Relative Percent Difference

SOP = Standard Operating Procedure

%R = Percent Recovery

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Aqueous**
Analytical Group	**Total Organic Carbon**
Analytical Method/ SOP Reference	**SW846 9060A / CA-763**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Aqueous**
Analytical Group	**Total Organic Carbon**
Analytical Method/ SOP Reference	**SW846 9060A / CA-763**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Control Sample	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Matrix Spike	One for every set 10 samples	%R must be within: 75-125	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Aqueous**
Analytical Group	**Total Organic Carbon**
Analytical Method/ SOP Reference	**SW846 9060A / CA-763**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Notes:

CA = Corrective Action

DL = Detection Limit

IDW = Investigation Derived Waste

LCS = Laboratory Control Sample

LOD = Limit of Detection

LOQ = Limit of Quantitation

QC = Quality Control

QSM = Quality Systems Manual

RPD = Relative Percent Difference

SOP = Standard Operating Procedure

%R = Percent Recovery

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Soil and IDW**
Analytical Group	**Ignitability**
Analytical Method/ SOP Reference	**SW846 1010A / CA-736**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil and IDW**
Analytical Group	**Ignitability**
Analytical Method/ SOP Reference	**SW846 1010A / CA-736**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
LCS	One per analytical batch of 20 or fewer samples.	Flash at 27ºC ±2 ºC	Investigate source of problem. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per ten samples	Results of sample and sample duplicate agree within ±2 °C – Report the lowest value.	If lab QC in criteria and duplicates do not agree within ±2 °C, report the lowest value and narrate the other values. Else, reanalyze	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Notes:

IDW = Investigation Derived Waste

LOD = Limit of Detection

LOQ = Limit of Quantitation

PQL = Practical Quantitation Limit

QC = Quality Control

SOP = Standard Operating Procedure

ºC = Degrees Celsius

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Soil and IDW**
Analytical Group	**pH**
Analytical Method/ SOP Reference	**SW-846 9045D/ CA-709**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
LCS	One per analytical batch of 20 or fewer samples.	%R must be ± 0.1 pH units	Investigate source of problem. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the PQL, <100% RPD for samples <3X the PQL.	Investigate problem and reanalyze sample in duplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Notes:
IDW = Investigation Derived Waste
LCS = Laboratory Control Sample
PQL = Practical Quantitation Limit
QC = Quality Control
RPD = Relative Percent Difference
%R = Percent Recovery

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**IDW**
Analytical Group	**Reactivity**
Analytical Method/ SOP Reference	**SW846 Chapter 7 / CA-733, CA-734**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**IDW**
Analytical Group	**Reactivity**
Analytical Method/ SOP Reference	**SW846 Chapter 7 / CA-733, CA-734**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
LCS	One per analytical batch of 20 or fewer samples.	Reactive Cyanide -%R must be within 0-100 Reactive Sulfide - %R must be within 50-150	Investigate source of problem. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	Reactive Cyanide -%R must be within 0-100 Reactive Sulfide - %R must be within 50-150	Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**IDW**
Analytical Group	**Reactivity**
Analytical Method/ SOP Reference	**SW846 Chapter 7 / CA-733, CA-734**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the PQL, <100% RPD for samples <3X the PQL.	Investigate problem and reanalyze sample in duplicate If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Notes:

IDW = Investigation Derived Waste

LCS = Laboratory Control Sample

PQL = Practical Quantitation Limit

QC = Quality Control

RPD = Relative Percent Difference

%R = Percent Recovery

SOP = Standard Operating Procedure

QAPP Worksheet #28a: Katahdin Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Solid**
Analytical Group	**Grain size**
Analytical Method/ SOP Reference	**ASTM D422 / CA-551**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
N/A: Laboratory QC samples are not planned for grain size analysis.						

Notes:

N/A = Not Applicable

QC = Quality Control

SOP = Standard Operating Procedure

QAPP Worksheet #28b: Eurofins TestAmerica Analytical Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

(EPA 2106-G-05 Section 2.3.5)

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Gamma Spec**
Analytical Method/ SOP Reference	**HASL 300 GA-01-R / ST-RD-0102**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method blank	One per preparation batch	No analytes detected > target detection limit	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Acceptable results per stated QC Acceptance Limits

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Gamma Spec**
Analytical Method/ SOP Reference	**HASL 300 GA-01-R / ST-RD-0102**

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
LCS	One per preparation batch	Recovery limits: 87-120% for Cs-137, 87-115% for Co-60, 87-116% for Am-241	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Acceptable QC Acceptance Limits
Duplicate	One per preparation batch	RPD limit of 40% or DER < 3	Correct problem, then re-prepare and reanalyze all samples in the associated preparatory batch, if not excursion not caused by sample matrix.	Analyst, Supervisor	Precision/ Accuracy/Bias	Acceptable QC Acceptance Limits

Notes:
DER = Duplicate Error Ration
LCS = Laboratory Control Sample
IDW = Investigation Derived Waste
MB = Method Blank
QC = Quality Control
RPD = Relative Percent Difference
SOP = Standard Operating Procedure

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Gross Alpha/Beta**
Analytical Method/ SOP Reference	**SW8463 9310 / ST-RD-0403**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Method blank	One per preparation batch	No analytes detected > target detection limit	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Supervisor	Accuracy/Bias/ Contamination	Acceptable results per stated QC Acceptance Limits
LCS	One per preparation batch	Recovery limits: 75-125% for Alpha; 75-125% for Beta	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Supervisor	Accuracy/Bias	Acceptable QC Acceptance Limits
Duplicate	One per preparation batch	RPD limit of 40% or DER < 3	Correct problem, then re-prepare and reanalyze all samples in the associated preparatory batch, if not excursion not caused by sample matrix.	Analyst, Supervisor	Precision/ Accuracy/Bias	Acceptable QC Acceptance Limits

Matrix	**Groundwater, Soil and IDW**
Analytical Group	**Gross Alpha/Beta**
Analytical Method/ SOP Reference	**SW8463 9310 / ST-RD-0403**

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Matrix Spike	One per preparation batch	Recovery limits: 43-123% for Alpha; 55-125% for Beta	Correct problem, then re-prepare and reanalyze all samples in the associated preparatory batch, if not excursion not caused by sample matrix.	Analyst, Supervisor	Accuracy/Bias	Acceptable QC Acceptance Limits

Notes:

DER = Duplicate Error Ration

LCS = Laboratory Control Sample

IDW = Investigation Derived Waste

MB = Method Blank

QC = Quality Control

RPD = Relative Percent Difference

SOP = Standard Operating Procedure

QAPP Worksheet #29: Project Documents and Records

(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Sample Collection and Field Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Field Logbook or Data Collection Sheets	Jerry Rawcliffe	Scott Calkin	Project File
Chain-of-Custody Forms	Jerry Rawcliffe	Bradley LaForest	Project File
Daily QC Reports	Jerry Rawcliffe	Julie Ricardi	Project File
Deviations	Jerry Rawcliffe	Scott Calkin	Project File
Corrective Action Reports	Julie Ricardi	Peter Baker	Project File
Correspondence	Peter Baker	Harry Hendler	Project File

Project Assessments			
Record	**Generation**	**Verification**	**Storage location/archival**
Field Audit Checklists	Julie Ricardi	Peter Baker	Project File
Data Verification Checklists	Bradley LaForest	Julie Ricardi	Project File
Data Validation Report	Bradley LaForest	Julie Ricardi	Project File
Data Usability Assessment Report	Bradley LaForest	Julie Ricardi	Project File

Laboratory Records			
Record	**Generation**	**Verification**	**Storage location/archival**
Sample Receipt and Login	Heather Manz	Bradley LaForest	Project File
Deviations	Heather Manz	Bradley LaForest	Project File
Corrective Action Reports	Heather Manz	Bradley LaForest	Project File

Laboratory Data Deliverables											
Record	VOC	SVOC	PCB	PCB Congeners	VPH	EPH	Metals	Radionuclides	pH	Grain Size	FOC
Narrative	X	X	X	X	X	X	X	X	X	X	X
COC	X	X	X	X	X	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X	X	X	X	X	X
QC Results	X	X	X	X	X	X	X	X	X		X
Chromatograms	X	X	X	X	X	X					
Raw Data	X	X	X	X	X	X	X	X	X	X	X

Notes:
COC = Contaminant of Concern

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action

(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Field Operations Readiness Review	Wood Quality Assurance Manager	Once	July 27, 2020	Readiness Review Checklist	24 hours following assessment

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Field Operations Readiness Review	Wood Field Operations Lead	Written response to Readiness Review findings	Immediately to within 24 hours of review	Wood Field Operations Lead	Wood Quality Assurance Manager

QAPP Worksheet #34: Data Verification and Validation Inputs

(UFP-QAPP Manual Section 5.2.1 and Table 9)

(EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	**X**	
2	Contract	**X**	
4	Field SOPs	**X**	
5	Laboratory SOPs	**X**	
Field Records			
6	Field logbooks	**X**	**X**
7	Equipment calibration records	**X**	**X**
8	Chain-of-Custody Forms	**X**	**X**
9	Sampling diagrams/surveys	**X**	**X**
10	Drilling logs	**X**	**X**
11	Geophysics reports	**X**	**X**
12	Relevant Correspondence	**X**	**X**
13	Change orders/deviations	**X**	**X**
14	Field audit reports	**X**	**X**
15	Field corrective action reports	**X**	**X**
Analytical Data Package			
16	Cover sheet (laboratory identifying information)	**X**	**X**
17	Case narrative	**X**	**X**
18	Internal laboratory chain-of-custody	**X**	**X**
19	Sample receipt records	**X**	**X**
20	Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	**X**	**X**

Item	Description	Verification (completeness)	Validation (conformance to specifications)
21	Communication records	X	X
22	Project-specific PT sample results	X	X
23	LOD/LOQ establishment and verification	X	X
24	Standards Traceability	X	X
25	Instrument calibration records	X	X
26	Definition of laboratory qualifiers	X	X
27	Results reporting forms	X	X
28	QC sample results	X	X
29	Corrective action reports	X	X
30	Raw data	X	X
31	Electronic data deliverable	X	X

Notes:

LOD/LOQ = Limit of Detection/Limit of Quantification

QAPP = Quality Assurance Project Plan

QC = Quality Control

SOP = Standard Operating Procedure

Data Verification = A check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records are available to proceed to data validation.
Data Validation = The evaluation of conformance to stated requirements, including those in the contract, methods, SOPs and the QPP.

Complete Chain-of-Custody information will be uploaded to FUDSChem within 5 days of sample collection.

QAPP Worksheet #35: Data Verification Procedures

(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbooks	QAPP, SOP S-6	Field notes will be reviewed periodically to determine completeness, appropriateness, ease of understanding, etc., of information recorded. Upon completion of field work, logbooks will be placed in the project files.	Project Manager or designee, Wood
Chain-of-custody forms	QAPP, SOP S-5	Chain-of-custody forms will be reviewed against the samples packed in the specific cooler prior to shipment. Original chain-of-custody forms will be sent with the samples to the laboratory, while a copy is retained for the project files.	Field Operations Lead or designee, Wood
Sample receipt and log-in acknowledgment forms	QAPP	Sample receipt and log-in summaries will be reviewed to determine potential receipt issues that may impact data quality and for consistency with the chain-of-custody forms.	Project Chemist, Wood Project Manager, Katahdin
Laboratory analytical data package prior to release	QAPP	Data packages and electronic deliverables will be reviewed/verified internally by the laboratory performing the work for completeness, adherence to QAPP requirements, and technical accuracy prior to submittal.	Project Manager, Katahdin
Laboratory deliverable	QAPP	Data packages will be reviewed by the Wood Project Chemist. The data will undergo Stage 2B/Stage 4 validation protocol.	Project Chemist, Wood
Data validation report	QAPP	Data validation reports will be reviewed by the Wood Project Manager and the Wood Quality Assurance Manager.	Project Manager and Quality Assurance Manager, Wood

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Electronic data	QAPP	Electronic laboratory data and field data will be reviewed for consistency with the hardcopy information.	Project Chemist, Wood
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Quality Assurance Manager, Wood

QAPP Worksheet #36 - Data Validation Procedures

(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)
Data Validator: Wood

Analytical Group/Method:	VOCs – SW846 8260	IDW VOCs – SW846 8260
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Results Only
Analytical specifications:	Worksheet 28 / SOP CA-202	Worksheet 28 / SOP CA-202
Measurement performance criteria:	Worksheet 12	NA
Percent of data packages to be validated:	100%	0%
Percent of raw data reviewed:	10%	0%
Percent of results to be recalculated:	10%	0%
Validation procedure:	EPA Region I Stage 4/Stage 2B	NA
Validation code (*see attached table):	S4VEM/S2BVEM	NV
Electronic validation program/version:	ADR	NA

Analytical Group/Method:	VPH – MA DEP VPH 04-1.1	VPH – MA DEP EPH 04-1.1
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Stage 4 Full Deliverable/ADR
Analytical specifications:	Worksheet 28 / CA-312	Worksheet 28 / CA-322
Measurement performance criteria:	Worksheet 12	Worksheet 12
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	10%	10%
Percent of results to be recalculated:	10%	10%
Validation procedure:	EPA Region I Stage 4/Stage 2B	EPA Region I Stage 4/Stage 2B
Validation code (*see attached table):	S4VEM/S2BVEM	S4VEM/S2BVEM
Electronic validation program/version:	ADR	ADR
Analytical Group/Method:	SVOCs – SW846 8270 SIM	PCBs / PCB Congeners – SW846 8082
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Stage 4 Full Deliverable/ADR
Analytical specifications:	Worksheet 28 / SOP CA-213	Worksheet 28 / SOP CA-329
Measurement performance criteria:	Worksheet 12	Worksheet 12
Percent of data packages to be validated:	100%	100%

Percent of raw data reviewed:	10%	10%
Percent of results to be recalculated:	10%	10%
Validation procedure:	EPA Region I Stage 4/Stage 2B	EPA Region I Stage 4/Stage 2B
Validation code (*see attached table):	S4VEM/S2BVEM	S4VEM/S2BVEM
Electronic validation program/version:	ADR	ADR

Analytical Group/Method:	IDW SVOCs – SW846 8270D	IDW PCBs – SW846 8082
Data deliverable requirements:	Result Only	Results Only
Analytical specifications:	SOP CA-226, CA-510	SOP CA-329
Measurement performance criteria:	NA	NA
Percent of data packages to be validated:	0%	0%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	Cursory Review	Cursory Review
Validation code (*see attached table):	NV	NV
Electronic validation program/version:	NA	NA

Analytical Group/Method:	IDW Pesticides – SW846 8081	IDW Herbicides – SW846 8151
Data deliverable requirements:	Result Only	Results Only
Analytical specifications:	SOP CA-302	SOP CA-305
Measurement performance criteria:	NA	NA
Percent of data packages to be validated:	0%	0%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	Cursory Review	Cursory Review
Validation code (*see attached table):	NV	NV
Electronic validation program/version:	NA	NA

Analytical Group/Method:	Metals – SW846 6020	IDW Metals - SW846 6010
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Results Only
Analytical specifications:	Worksheet 28 / SOP CA-627	SOP CA-608
Measurement performance criteria:	Worksheet 12	NA

Percent of data packages to be validated:	100%	0%
Percent of raw data reviewed:	10%	0%
Percent of results to be recalculated:	10%	0%
Validation procedure:	EPA Region I Stage 4/Stage 2B	Cursory Review
Validation code (*see attached table):	S4VEM/S2BVEM	NV
Electronic validation program/version:	ADR	NA

Analytical Group/Method:	Metals – SW846 7471	IDW Metals – SW846 7470
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Results Only
Analytical specifications:	Worksheet 28 / SOP CA-611	SOP CA-615
Measurement performance criteria:	Worksheet 12	NA
Percent of data packages to be validated:	100%	0%
Percent of raw data reviewed:	10%	0%
Percent of results to be recalculated:	10%	0%
Validation procedure:	EPA Region I Stage 4/Stage 2B	Cursory Review
Validation code (*see attached table):	S4VEM/S2BVEM	NV
Electronic validation program/version:	ADR	NA

Analytical Group/Method:	Radionuclides Gross Alpha/Beta – SW846 9130	Radionuclides Gamma Spec – GA-01-R
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Stage 4 Full Deliverable/ADR
Analytical specifications:	Worksheet 28 / SOP ST-RD-0403	Worksheet 28 / SOP ST-RD-0102
Measurement performance criteria:	Worksheet 12	Worksheet 12
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	10%	10%
Percent of results to be recalculated:	10%	10%
Validation procedure:	EPA Region I Stage 4/Stage 2B	EPA Region I Stage 4/Stage 2B
Validation code (*see attached table):	S4VEM/S2BVEM	S4VEM/S2BVEM
Electronic validation program/version:	ADR	ADR

Analytical Group/Method:	pH – SW846 9045	IDW pH-Corrosivity – SW846 9045
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Results Only
Analytical specifications:	Worksheet 28 / SOP CA-709	Worksheet 28 / SOP CA-709
Measurement performance criteria:	Worksheet 12	NA
Percent of data packages to be validated:	100%	0%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	EPA Region I Stage 2B	Cursory Review
Validation code (*see attached table):	S2BVEM	NV
Electronic validation program/version:	ADR	NA

Analytical Group/Method:	Grain Size – ASTM D422	FOC – SW846 9060A
Data deliverable requirements:	Stage 4 Full Deliverable/ADR	Stage 4 Full Deliverable/ADR
Analytical specifications:	Worksheet 28 / SOP CA-551	Worksheet 28 / SOP CA-741
Measurement performance criteria:	Worksheet 12	Worksheet 12
Percent of data packages to be validated:	100%	100%
Percent of raw data reviewed:	0%	10%
Percent of results to be recalculated:	0%	10%
Validation procedure:	EPA Region I Stage 2B	EPA Region I Stage 2B
Validation code (*see attached table):	S2BVEM	S2BVEM
Electronic validation program/version:	ADR	ADR

Analytical Group/Method:	IDW Cyanide – SW846 9012	IDW Sulfide – SW846 9034
Data deliverable requirements:	Results Only	Results Only
Analytical specifications:	Worksheet 28 / SOP CA-733	Worksheet 28 / SOP CA-734
Measurement performance criteria:	NA	NA
Percent of data packages to be validated:	0%	0%
Percent of raw data reviewed:	0%	0%
Percent of results to be recalculated:	0%	0%
Validation procedure:	Cursory Review	Cursory Review
Validation code (*see attached table):	NV	NV

Electronic validation program/version:	NA	NA

Analytical Group/Method:	IDW Ignitability – SW846 1010 Modified	
Data deliverable requirements:	Results Only	
Analytical specifications:	Worksheet 28 / SOP CA-736	
Measurement performance criteria:	NA	
Percent of data packages to be validated:	0%	
Percent of raw data reviewed:	0%	
Percent of results to be recalculated:	0%	
Validation procedure:	Cursory Review	
Validation code (*see attached table):	NV	
Electronic validation program/version:	NA	

Validation Code and Label Identifier Table (To be attached to the QAPP)

Validation Code*	Validation Label	Description/Reference
S2BVEM	Stage 2B Validation Electronic and Manual	EPA 540-R-08-005
S4VEM	Stage 4 Validation Electronic and Manual	EPA 540-R-08-005
NV	Not Validated	EPA 540-R-08-005

Wood chemists will perform validation on field sample data in accordance with the table above and following the USEPA National Functional Guidelines for Organic and Inorganic Superfund Methods (USEPA, 2017), with QC criteria presented in **Worksheet #12**, **Worksheet #24**, and **Worksheet #28**. Professional judgment of the data validators will be used during review of data.

The following qualifiers may be assigned during the validation process. Potential impacts on DQOs will be discussed in the data validation report.

J – estimated concentration
J- – estimated concentration, result biased low
J+ – estimated concentration, result biased high
UJ – not detected and the detection limit is estimated
U – not detected
NJ – the analyte has been tentatively identified as present and the concentration is estimated

R – rejected (note final rejection of data and their use is a decision reserved for the project team). X qualifiers will be automatically applied in FUDSChem if applicable. If the PDT decides that the data should be rejected the final qualifier will be changed to R.

The following data validation guidelines will be followed for sample reporting and blank detections:

- The general reporting convention will be to report non-detected results at the LOD, with detections reported down to the MDL. Detections less than the LOQ will be reported by the laboratory with J qualifiers.
- Sample results with associated blank contamination will be qualified as follows:

Method Type	Blank Result	Sample Result	Action
Organics and Wet Chemistry	< LOQ	< LOD	Qualify non-detect (U) at LOD
		LOD < Sample Result < LOQ	Qualify non-detect (U) at reported result
		≥ LOQ	≤ 2x blank: Qualify non-detect (U) at reported result. ≥ 2x blank: no qualification
	≥ LOQ	< LOD	Qualify non-detect (U) at LOD
		LOD < Sample Result < LOQ	Qualify non-detect (U) at reported result
		≥ LOQ and < blank result	Qualify non-detect (U) at reported result
		≥ LOQ and ≥ blank result	≤ 2x blank: Qualify non-detect (U) at reported result. 2x-5x blank: J+
Metals	≤ LOQ	≤ LOD and < 10x blank result	Qualify non-detect (U) at LOD.
		LOD < Sample Result ≤ LOQ and < 10x blank result	Qualify non-detect (U) at reported result.
		> LOQ	< 10x blank result; qualify estimated (J+)
	> LOQ	≤ LOD	Qualify non-detect (U) at LOD
		LOD < Sample Result ≤ LOQ	Qualify non-detect (U) at reported result
		> LOQ but < 10x blank result	Qualify estimated (J+)
		≥ 10x blank result	No qualification

QAPP Worksheet #37: Data Usability Assessment

(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

The complete PDT responsible for participating in the data usability assessment:

- Wood and USACE Project Manager
- Wood and USACE Quality Assurance Manager
- Wood and USACE Ecological Risk Assessor
- Wood and USACE Human Health Risk Assessor
- Wood and USACE RI Technical Lead
- Wood and USACE Project Chemist
- Wood and USACE Biologist
- Wood Field Operations Lead

The data usability assessment will be performed at the conclusion of data collection activities, using the outputs from data verification and data validation (Worksheets #34, #35, #36). The data usability assessment involves a qualitative and quantitative evaluation of the data to determine if the data are of the right type, quality, and quantity to support the decisions that need to be made, and whether the results can be used as intended with an acceptable level of confidence.

The quality and usability of data obtained during the project will be determined by reviewing and inspecting field logbooks, sampling forms, chain of custody forms, laboratory data packages, and data validation reports; and by verifying that the sampling procedures and analytical results were obtained following the applicable protocols such that they will satisfy project requirements and can be relied upon for evaluating the data with respect to project DQOs. The data usability assessment will identify possible effects on data usage resulting from project requirement failures (data quality), and the adequacy of the data in meeting project-specific QA/QC requirements (data usability).

Efforts to evaluate and verify attainment of project requirements will enable data users to understand usability limitations associated with project data. Procedures used to assess QA/QC objectives will be in accordance with the analytical methods, which were selected based on the method's ability to meet project goals.

The data usability assessment, consisting of data quality/usability and project DQO reconciliation evaluations, will be performed by personnel with the appropriate training and/or experience to perform the reviews/evaluations and will include Wood personnel listed above, as appropriate.

The data usability assessment (DUA) will be conducted using a 5-step process consisting of:

1. Review of DQOs and data collection documentation to assess compliance with this QAPP.

2. Review data validation reports to assess analytical data quality as defined by this QAPP. Review deviations from planned activities and assess data gaps, if any. Evaluate implications of unacceptable QC results.
3. Compare analytical results, including detection limits, to potentially applicable screening values. Look for patterns, trends, and anomalies in the data.
4. Based on the above, determine whether the data meet project DQOs and assess impact on data usability.
5. Draw conclusions from the data and evaluate against existing CSM and project approach to determine if the CSM or project approach needs to be adjusted, or if additional evaluation of the data is required to address questions. Prepare the data usability summary report.

The PDT will assess the following data quality indicators during data validation and/or data usability assessment:

Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions. Usually, combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically as a relative percent difference (RPD).

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

Where:

A = First duplicate concentration

B = Second duplicate concentration

Field sampling precision is evaluated by analyzing field duplicate samples. Field duplicates will be collected and analyzed at a frequency of five percent for samples collected. Field duplicate precision will be evaluated against performance criteria presented in **Worksheet #12.**

Laboratory analytical precision is evaluated by analyzing laboratory duplicates or MS/MSD. MS/MSD samples will be generated for all analytes for this project. The results of the analysis of each MS/MSD pair will be used to calculate an RPD for evaluating precision. Laboratory duplicate precision will be evaluated against performance criteria presented in **Worksheet #12.**

Accuracy/Bias

Analytical bias derived from field activities will be assessed by collecting and analyzing field and equipment blanks as appropriate. These QC samples will be used to evaluate the potential for target analytes to enter samples as a result of sampling processes. Bias introduced in the field will be evaluated against performance criteria presented in **Worksheet #12.**

A program of sample spiking will be conducted to evaluate laboratory accuracy/bias. This program includes analysis of the MS and MSD samples, laboratory control sample (LCS), surrogate spikes, labeled isotope dilution compounds, and laboratory blanks. MS and MSD samples will be collected, prepared, and analyzed at a frequency of five percent per matrix. LCS (for applicable methods) will be analyzed with every batch containing field samples. Surrogate standards or labeled isotope dilution compounds (for applicable methods) will be added to every field and QC sample analyzed. The results of the spiked samples will be used to calculate the percentage of recovery to evaluate accuracy. Accuracy and bias will be evaluated against performance criteria presented in **Worksheet #12.**

$$\%R = \frac{S - C}{T} \times 100$$

Where:

S = Measured spike sample concentration

C = Sample concentration

T = True or actual concentration of the spike

Results that fall outside the accuracy goals will be further evaluated based on the results of other QC samples.

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this QAPP, and when none of the QC criteria that affect data usability is exceeded. When all data validation is completed, the number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with a rejected (R) flag. The requirement for completeness is 95% for results and is determined using the following equation:

$$\%Completeness = 100 \times \frac{\text{number of valid analyte results}}{\text{number of possible results}}$$

At completion of each sampling event and after receipt of final laboratory data packages, the completeness of the data will be assessed. If data omissions are identified, the associated sample may be re-sampled and/or reanalyzed, if feasible. Laboratory results will be reviewed as they become available to assess laboratory performance and its effect on data completeness requirements.

Comparability

Comparability expresses the confidence with which data from one sample, sampling round, site, laboratory, or project can be compared to those from another similar data source. Comparability during sampling is dependent upon sampling program design. Comparability during analysis is dependent upon analytical methods, detection limits, laboratories, units of measure, and sample preparation procedures. Comparability is determined on a qualitative rather than quantitative basis. For this project, comparability of data collected will be ensured by adherence to standard sample collection procedures, standard field measurement procedures, and standard reporting methods, including consistent units. For example, concentrations will be reported in a manner consistent with general industry practice (soil data will be reported on a dry-weight basis). In addition, to support the comparability of fixed-base laboratory analytical results with those obtained from previous or future testing, all samples will be analyzed by USEPA-approved methods. The USEPA-recommended maximum permissible sample holding times (**Worksheet #19**) for organic and inorganic parameters will not be exceeded. Instrument calibrations will be performed in accordance with USEPA method specifications and will be checked at the frequency specified for the methods. As needed, performance evaluation samples may be added to the analytical program to ensure data comparability. Performance evaluation sample recoveries will be compared against the manufacturer's specified recovery limits or 60 to 140 percent recovery limits, if there are no manufacturer's specified recovery limits.

Representativeness

Representativeness expresses the extent to which collected data characterize the presence or absence of contaminants at a given location. Sample collection, handling, preservation, and analytical procedures are designed to obtain the most representative sample possible. Representative samples will be achieved by the following:

- Collection of samples from locations representing site conditions.
- Use of appropriate sample preservation techniques.
- Use of appropriate sampling procedures, including proper equipment;
- Use of appropriate analytical methods for the required parameters; and,
- Analysis of samples within the required holding times.

Sample representativeness is also affected by the portion of each sample chosen for analysis. The laboratory will adequately homogenize all samples prior to taking aliquots for analysis to ensure that the reported results are representative of the sample received.

Sensitivity

The concentration of any one target compound that can be detected and/or quantified is a measure of sensitivity for that compound. Sensitivity is instrument-, compound-, method-, and matrix-specific. The subcontract laboratory will flag (as an estimate, "J" flag) and report target compounds detected below the LOQ down to the DL in an effort to meet project DQOs.

The data usability assessment, including verification and validation procedures employed during this project, will document whether data collected meet project DQOs and assure a reasonable basis for decision making.

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Appendix A

Field Standard Operating Procedures

SOP No. S-1

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

SURFACE SOIL SAMPLING

wood.

STANDARD OPERATING PROCEDURE

SURFACE SOIL SAMPLING

1.0 SCOPE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used for obtaining surface soil samples for physical or chemical analysis. Collection of soil samples for laboratory analysis for volatile organic compounds may require specially prepared containers, syringes, or Encore samplers. This SOP also describes the procedures for using the various types of sampling equipment, which include shovels, trowels, and hand-augers. The equipment may be constructed of special materials (for example, stainless steel, inert plastics) according to specific project requirements.

2.0 RESPONSIBILITIES

The field sampling personnel will be responsible for the proper use and maintenance of all types of equipment used for obtaining surface soil samples, and the collection, labeling, handling and storage of all samples until further chain of custody procedures are undertaken.

3.0 EQUIPMENT AND SUPPLIES

3.1 Shovel - Long or Short Handle Type

Used for penetrating the upper surface and/or obtaining soil samples directly. Shovel blade must not be painted when obtaining samples for metals analysis.

3.2 Trowel - Basic Garden Variety, which Resembles a Small Shovel

Constructed of steel or polypropylene (plastic). The blade of a trowel is generally flat and 5 to 6 inches in length. A scoop (blade has curved edges versus flat) may be substituted if necessary.[1] The trowel blade must not be painted when sampling for metals analysis.

3.3 Hand Augers

These tools are generally comprised of a short, hollow, thin-walled augers connected to a "T" shaped handle. Clockwise rotation of the T-handle with moderate downward pressure initiates the cutting and soil sampling process. Some augers are designed to accommodate an optional, plastic or metal, cylindrical sample sleeve which can be inserted into the body of the auger to facilitate sample collection and to avoid cross-contamination. The use of sampling sleeves is not necessary if adequate decontamination is performed between sampling locations and or depths (unless they are specified in

[1] Requirements for inert materials, decontamination, or calibrated sampling tools may be required depending upon the purpose of the sampling. These requirements will be detailed in a project-specific sampling plan.

a work plan). If laboratory samples are not being collected, sample sleeves are usually not used.

3.4 Sample Containers

Select appropriate sample containers depending upon the analyses to be performed as described in the project Sampling and Analysis Plan (SAP) and Quality Assurance Project Plan (QAPP).

3.5 Field Logbook and Surface Soil Field Data Record

Field data should be recorded on the Soil Sampling Field Data Record (See SAP). These reports along with any analytical data will be provided in some form or investigation or completion report.

4.0 PROCEDURES

4.1 General

Specific sampling equipment and methodology will be dictated by the characteristics of the soil to be sampled, field conditions, the type of soil samples required by the project, and the analytical procedures to be employed. Soil samples obtained from the near-surface (0-2 feet below the ground surface) may be collected using a shovel or trowel. The type of analysis required (e.g., grain-size distribution, physical, chemical) may require specific soil amounts or the use of specialized sampling equipment.

Sampling locations or sampling design will be identified in the SAP.

A hand-auger can be used to extract shallow soil samples from depths as deep as three to four feet below the surface. Representative samples are collected directly from the auger flight as it is withdrawn from the ground, or from the tube sampler attached to the end of the rods. Levels of personal protection will be described in the Site Safety and Health Plan (SSHP).

4.2 Detailed Procedures

Select the Specific Sampling Location in Accordance with the Project Sampling Plan. Construct a sampling grid if necessary. Begin recording information on the Surface Soil Field Data Record. Prepare the sampling location by removing all surface materials that are not to be included in the sample (i.e., rocks, twigs, and leaves).

Select the Previously Decontaminated Type of Sampler Required to Obtain the Correct Sample. At the surface, use a shovel, trowel, or tube sampler; below surface, use a hand-auger or tube sampler.

Remove decontamination seals (i.e., aluminum foil/plastic wrap).

Obtain a sufficient quantity of soil for the desired chemical or physical analyses. If volatile organic compound (VOC) or volatile petroleum hydrocarbon (VPH) samples are scheduled, they should be collected immediately in accordance with SOP No. S-8 Field Preservation of volatile organic analysis (VOA) and VPH Soil Samples (See SAP). The remaining soil should then be composited in a stainless steel bowl for all other analytical parameters.

When using the hand-auger, advance the auger to the required depth, then slowly remove the auger and collect the soil sample from the auger flight at the point corresponding to the required depth. Re-insert and continue augering if deeper samples are required. If samples are required from sandy or

non-cohesive soil, use of a hand trowel or shovel may be necessary. Soil samples obtained directly from auger flights are, at best, composite samples over a portion of the auger hole. Samples should only be taken from auger-flights when composite samples are desired. Select the appropriate sample container and place the sample in the container. Describe the soil in accordance with SOP No. S-7 Procedure for Description and Identification of Soils (See SAP).

Cap and label the sample container. Record all observations on the Surface Soil Sampling Field Data Record Form and field logbook. Place samples into a cooler and begin specified storage and preservation procedures.

Decontaminate the sampler between collection points. Decontamination procedures will be performed as identified in SOP S-3, Decontamination of Field Equipment.

Initiate delivery of the samples to the designated Field Operations Leader or sample manager.

5.0 DOCUMENTATION

Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms include:

1. Field Logbooks

2. Surface Soil Sampling Field Data Record

6.0 QUALITY CONTROL/QUALITY ASSURANCE

Quality control for this groundwater sampling method involves the collection of field quality control samples including field duplicated, matrix spike/matrix spike duplicate samples, trip blank samples, equipment blank samples, and temperature blank samples. Frequency of collection or QC samples will be specified in the SAP.

7.0 REFERENCES

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SOP No. S-2

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
CALIBRATION OF FIELD INSTRUMENTS FOR WATER QUALITY PARAMETERS

STANDARD OPERATING PROCEDURE

CALIBRATION OF FIELD INSTRUMENTS FOR WATER QUALITY PARAMETERS

1.0 SCOPE AND APPLICABILITY

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for groundwater and surface water. Water quality instruments addressed in this SOP include those that measure temperature, pH, dissolved oxygen (DO), conductivity/specific conductance, oxidation-reduction potential (ORP), and turbidity.

This SOP is written for instruments that utilize multiple probes for temperature, pH, DO, conductivity/specific conductance, ORP, and turbidity. This SOP refers to instrumentation and outlines calibration procedures consistent with those discussed in U.S. Environmental Protection Agency (USEPA) Region I Standard Operating Procedure, Calibration of Field Instruments, 23 March 2017; Revision 3.

For groundwater monitoring during well development and/or purging prior to sample collection, the multiple probe instrument must be equipped with a flow-through cell, and the display/logger or computer display screen should be large enough to simultaneously display the readouts of each probe in the instrument. Turbidity is measured using a separate instrument because turbidity cannot be measured accurately in a flow-through cell.

2.0 SUMMARY OF METHOD

All monitoring instruments must be calibrated before they are used to measure environmental samples. Most instruments will require at least two standards to bracket the expected measurement range, one standard less than the expected value and one higher. At a minimum, calibration must be performed at the beginning of each sampling day prior to sample collection. Site-specific plans should be consulted for required calibration frequency. Note: Part of the instrument preparation and initial calibration is performed prior to the field event.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

3.0 HEALTH & SAFETY WARNINGS

Wood Environment & Infrastructure Solutions, Inc. (Wood) employees will be on site when implementing this SOP. Therefore, Wood personnel shall follow the Site Safety and Health Plan (SSHP). Wood personnel will use the appropriate level of personal protective equipment (PPE), which includes the following:

1) hardhat;

2) safety boots (steel toe/steel shank);

3) safety glasses; and

4) chemical resistant gloves.

Implementing this SOP will require the use of calibration solutions. The following health and safety precautions must be taken with the pH, conductivity, and ORP solutions: Avoid inhalation, skin and eye contact or ingestion.

Maintenance of the instruments will require the use of liquid cleaners. Although these substances are not hazardous materials, Wood will appropriately handle and store them at times in accordance with manufacturer's instructions.

4.0 CAUTIONS & POTENTIAL PROBLEMS

Prior to calibration all instrument probes must be cleaned according to the manufacturer's instructions. Failure to perform this step (proper maintenance) can lead to erroneous measurements.

Prior to using calibration standards, check all expiration dates.

Use a ring stand and clamp to secure the sonde in an upright position. This will prevent the sonde from falling over and damaging the probes.

The volume of the calibration solutions must be sufficient to cover both the probe being calibrated and the temperature sensor (see manufacturer's instructions for additional information).

While calibrating or performing sample measurements, make sure there are no air bubbles lodged between the probe and the probe guard.

DO content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic and or erroneous measurements. If the probe reading shows the error message "value out of range", the instrument probe must be recalibrated.

5.0 PERSONNEL QUALIFICATIONS

Since this SOP will be implemented at sites or in work areas that entail potential exposure to toxic chemicals or hazardous environments, all Wood personnel must be adequately trained.

Before implementing this SOP alone, Wood personnel must be trained in these procedures by a senior staff member with experience operating the equipment. In addition, all personnel utilizing this SOP must have completed the following:

- 40-hour OSHA training;
- 8-hour annual refresher training; and
- On-site training.

In addition to the 40-hour initial OSHA; training (and annual 8-hour refresher training), all Wood field staff will complete 24 hours of supervised field experience that contribute toward the 24-hour field supervised requirement in compliance with OSHA regulation: 29 CFR 1910.120(e)(4).

6.0 EQUIPMENT AND SUPPLIES

The following equipment should be used when calibrating water quality parameter measuring equipment. Site-specific conditions may warrant the use of additional items or deletion of items from this list.

- Appropriate level of personal protection
- Water quality meter capable of measuring pH, temperature, DO, specific conductivity, and ORP (e.g., YSI 600XL, or equivalent)
- Turbidity Meter (e.g., LaMotte 2020, or equivalent)
- Distilled water
- Deionized water
- Flow-through cell
- Ring stand with clamp
- Paper towels
- Soft tissue (e.g., Kimwipes)
- Cuvette
- pH buffer solutions (4, 7, 10)
- Conductivity solution (100, 1000 µmhos)
- Zobell solution
- Turbidity standards (0.5, 20 NTU)
- Zero DO solution (0.0 milligrams per liter [mg/L])
- DO membrane kit (electrolyte solution, membranes)
- NIST thermometer (0.01 C accuracy)
- Small glass or polyethylene jars to hold the calibration standards (4-8 oz.)
- Calibration Logbook
- Field Instrument Calibration Field Data Record (See Sampling and Analysis Plan)
- Cup or spray bottle for the distilled water

7.0 PROCEDURES

The probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature by the instrument. Communications to the instrument (programming and displaying the measurement files) are performed using a display/logger or a computer. Information sent to the instrument is entered through the keypad on the display/logger or computer. It is desirable that the display/logger or computer have data storage capabilities. If the instrument does not have a keypad, follow the manufacturer's instructions for entering information into the instrument.

- Program the multi-probe instrument so that the following parameters to be measured will be displayed: temperature, pH, percent DO, mg/L dissolved oxygen, conductivity, specific conductance, and ORP.
- For instrument probes that rely on the temperature sensor (pH, DO, conductivity/specific conductance, and ORP), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions.

Temperature

Most instrument manuals state that calibration of the temperature sensor is not required, but this SOP requires that the temperature sensor be checked to verify its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the instrument. If the accuracy check date/information is not included with the instrument or the last check was performed over a year prior to the date of use, it is recommended that the temperature sensor accuracy be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked.

8 VERIFICATION PROCEDURE

1. Allow a container filled with water to equilibrate to ambient temperature.
2. Place a NIST -traceable thermometer and the instrument's temperature sensor into the water and wait approximately five minutes for both temperature readings to stabilize.
3. Compare the two measurements. The instrument's temperature sensor must agree with the NIST - traceable thermometer measurement within the accuracy of the sensor (usually to +/-15°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

Dissolved Oxygen

DO is the volume of oxygen that is dissolved in water and is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic or erroneous measurements.

9.0 CALIBRATION PROCEDURE

1. Gently dry the temperature sensor according to manufacturer's instructions.
2. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container that comes with the instrument.
3. Place the DO probe in the container without the probe coming in contact with the wet sponge or paper towel. The probe must fit loosely in the container to ensure it is vented to the atmosphere.
4. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn on the instrument to allow the DO probe to warm up. Select monitoring/run mode. Check temperature readings. Readings must stabilize before continuing to the next step.
5. Select calibration mode; then select "DO%".
6. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement can be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location and unless this is the only source of barometric data. [Note: inches of mercury times 25.4 mm/inch mercury equals mm of mercury].
7. The instrument should indicate that the calibration is in progress. After calibration, the instrument should display percent saturated DO. Check the reading against the Temperature Atmospheric Pressure table in Attachment A. For example, if the barometric pressure is 752 mm Hg at an elevation of 278 feet, the percent saturation value after calibration should be 99%.
8. While the probe is still in the calibration cup, select monitoring/run mode. Compare the DO mg/L reading to the Oxygen Solubility at Indicated Pressure chart in Attachment A. For example, if the barometric pressure is 750 mm Hg and the temperature inside the calibration cup is 20°C, the DO mg/L reading should be 8.94 mg/L. If they do not agree to the accuracy of the instrument (usually ±0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution and repeat calibration. If this does not work, change the membrane and electrolyte solution and repeat calibration.
9. Remove the probe from the container, rinse it with distilled water, pat it dry with a towel and place it into a 0.0 mg/L DO Standard. The standard must be filled to the top of its container and the DO probe must fit snugly into the standard's container (no headspace). Check temperature readings. They must stabilize before continuing.
10. Wait until the "mg/L DO" readings have stabilized. The instrument should read < 0.5 mg/L or to the accuracy of the instrument (usually ± 0.2 mg/L) within 30 seconds. If the instrument cannot reach this value, it will be necessary to clean the probe and change the membrane and electrolyte solution. If this does not work, prepare a new mg/L standard.

11. If these measures do not work, contact the manufacturer.

pH (electrometric)

The pH is the measure of the degree of the acidity or alkalinity of a solution as measured on a scale of 0 to 14. The pH of a sample is determined electrometrically using a glass electrode. All pH measurements are in standard units (SU).

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. For groundwater, the pH will usually be close to seven. Three standards are needed for the calibration: one close to seven, one at least two pH units below seven and the other at least two pH units above seven. For those instruments that will not accept three standards, the instrument will need to be recalibrated if the water sample's pH is outside the range defined by the two standards used in the initial calibration.

10.0 CALIBRATION PROCEDURE

1. Allow the buffered standards to equilibrate to the ambient temperature.
2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.
3. Remove the cover of the probe, rinse in a cup filled with distilled water or use a spray bottle, and blot dry with soft tissue.
4. Select monitoring/run mode. Immerse probe in the initial buffered standard (e.g., pH 7) and allow at least 1 minute for temperature equilibration before proceeding.
5. Enter the buffered standard value (7) into the pH calibration menu of the instrument. Allow the pH reading to stabilize for approximately 30 seconds and if the reading does not change, finish the calibration. The reading should remain within the manufacturer's specifications; if it changes, recalibrate. If readings continue to fluctuate or readings do not stabilize after recalibration, consult the manufacturer.
6. Remove probe from the initial buffered standard, rinse in a cup filled with distilled water or use a spray bottle, and blot dry with soft tissue.
7. Immerse probe into the second buffered standard (e.g., pH 4). Repeat step 5 substituting "4" into the pH calibration menu instead of "7".
8. Remove probe from the second buffered standard, rinse in a cup filled with distilled water or use a spray bottle, and blot dry with soft tissue. If the instrument only accepts two standards the calibration is complete. Proceed to step 11. Otherwise continue with step 9.

9. Immerse probe m third buffered standard (e.g., pH 10). Repeat step 5, substituting "10" into the pH calibration menu instead of "7".

10. Remove probe from the third buffered standard, rinse in a cup filled with distilled water or use a spray bottle, and blot dry with soft tissue.

11. Select monitoring/run mode, if not already selected. To ensure that the initial buffered calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the reading to stabilize. The reading should read the initial standard value (e.g., 7) within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

Specific Conductance

Conductivity is used to measure the ability of an aqueous solution to conduct an electrical current. Specific conductance is the conductivity value corrected to 25°C. Calibrating an instrument for specific conductance automatically calibrates the instrument for conductivity, and vice-versa.

Most instruments are calibrated against a single standard which is near, but below the specific conductance of the environmental samples. A second standard which is above the environmental sample specific conductance is used to check the linearity of the instrument in the range of measurements.

10.0 CALIBRATION PROCEDURE

1. Allow the calibration standard to equilibrate to the ambient temperature.
2. Remove probe from its storage container, rinse the probe with a small amount of the conductivity/specific conductance standard (discard the rinsate), and place the probe into the conductivity/specific conductance standard. Gently move the probe up and down in the solution to remove any air bubbles from the sensor. Allow the probe to sit in the solution for at least l minute for temperature equilibration before proceeding.
3. Select calibration mode.
4. Select Specific Conductance from the Calibration menu. Enter the calibration value of the solution (mS/cm at 25°C) and continue. Allow the Specific Conductance reading to stabilize for approximately 30 seconds and finish the calibration. The reading should remain within manufacturer's specifications. If it does not, recalibrate. If readings continue to change after recalibration, consult the manufacturer.
5. Remove probe from the standard, rinse the probe with a small amount of the second conductivity/specific conductance standard (discard the rinsate), and place the probe into the second conductivity/specific conductance standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer, but

above the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

NOTE: These procedures should only be used for instruments that are capable of automatically correcting specific conductance for temperature (to 25°C). For instruments that cannot calibrate for specific conductance, follow the procedures in the instrument's manual for conductivity calibration. If calibrating for conductivity instead of specific conductance, the solutions conductivity value must be corrected for the temperature that the sensor is reading.

Oxidation-Reduction Potential

The ORP is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts (mV) and is temperature dependent.

11.0 CALIBRATION OR VERIFICATION PROCEDURE

1. Allow the calibration standard (Zobell Solution) to equilibrate to ambient temperature.
2. Remove the cover of the probe and place it into the standard.
3. Select monitoring/run mode.
4. While stirring the standard, wait for the probe temperature to stabilize, and then read the temperature.
5. Look up the mV value at this temperature from the mV versus temperature correction table found in Attachment C. It may be necessary to interpolate mV values between temperatures. Select "calibration mode", then "ORP". Enter the temperature corrected ORP value and calibrate the instrument.
6. Select monitoring/run mode. The reading should remain unchanged within manufacturer's specifications. If it changes, recalibrate. If readings continue to change after calibration, consult manufacturer.
7. If the instrument instruction manual states the instrument is factory calibrated, then verify the factory calibration against the standard. If reading does not agree within the specification of the instrument, the instrument will need to be re-calibrated by the manufacturer.

Turbidity:

Turbidity refers to how clear the water is and is a measure of relative sample clarity. The greater the amount of total suspended solids in the water, the higher the measured turbidity. The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A turbidity meter is a nephelometer with a visible light source for illuminating the sample and one or more photoelectric detectors placed ninety degrees to the path of the light source.

Some instruments will only accept one standard. For these instruments, the standards will serve as check points.

12.0 CALIBRATION PROCEDURES

1. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.
2. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry, free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette, respectively.
3. Zero the instrument by using either a zero or 0.02 NTU standard. A zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.
4. Using a standard at I NTU, calibrate according to manufacturer's instructions or verify calibration if instrument will not accept a second standard. If verifying, the instrument should read the standard value to within the specifications of the instrument. If the instrument has a range of scales, check each range that will be used during the sampling event with a standard that falls within that range.
5. Using a standard at 10 NTU, calibrate according to manufacturer's instruction or verify calibration if instrument does not accept a third standard. If verifying, the instrument should read the standard value to within the specifications of the instrument.

Note: If only performing a two-point calibration (depending on project requirements), the 0.02 NTU and 10 NTU standard should be used.

12.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

Prior to calibrating, the field equipment and calibration standard information should be recorded on a separate Field Instrument Calibration Record (See SAP). For field equipment, the information recorded should include the make, model number and the serial number of the instrument. Each instrument can be assigned an identification number which can be referenced in future field notes or when filling out the Field Instrument Calibration Field Data Record.

For calibration standards, the information recorded should include the manufacturer, expiration date, true value, and standard description such as lot number. Each calibration standard can also be assigned an identification number which can be referenced in future field notes or when filling out the Field Instrument Calibration Record.

All standards should be initialed and dated when opened.

At a minimum, the log must include the instrument information described above, calibration standard information described above, calibration date and time, and the instrument calibration results.

13.0 REFERENCES

USEPA Region I, March 23, 2017. Standard Operating Procedure; "Calibration of Field

Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)"; March 23, 2017; Revision 3.

USEPA Region I, July 30, 1996. Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples for Monitoring Wells.

Attachment A

Dissolve Oxygen – Oxygen Solubility Table

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Dissolved Oxygen Tables

CALIBRATION AND OXYGEN SOLUBILITY TABLES

Calibration Table Calibration Values for Various Atmospheric Pressures and Altitudes

PRESSURE			ALTITUDE		CALIBRATION VALUE
Inches Hg	mm Hg	kPa	Feet	Meters	Percent Saturation
30.23	768	102.3	-276	-84	101
29.92	760	101.3	0	0	100
29.61	752	100.3	278	85	99
29.33	745	99.3	558	170	98
29.02	737	98.3	841	256	97
28.74	730	97.3	1126	343	96
28.43	722	96.3	1413	431	95
28.11	714	95.2	1703	519	94
27.83	707	94.2	1995	608	93
27.52	699	93.2	2290	698	92
27.24	692	92.2	2587	789	91
26.93	684	91.2	2887	880	90
26.61	676	90.2	3190	972	89
26.34	669	89.2	3496	1066	88
26.02	661	88.2	3804	1160	87
25.75	654	87.1	4115	1254	86
25.43	646	86.1	4430	1350	85
25.12	638	85.1	4747	1447	84
24.84	631	84.1	5067	1544	83
24.53	623	83.1	5391	1643	82
24.25	616	82.1	5717	1743	81
23.94	608	81.1	6047	1843	80
23.62	600	80.0	6381	1945	79
23.35	593	79.0	6717	2047	78
23.03	585	78.0	7058	2151	77
22.76	578	77.0	7401	2256	76
22.44	570	76.0	7749	2362	75
22.13	562	75.0	8100	2469	74
21.85	555	74.0	8455	2577	73
21.54	547	73.0	8815	2687	72
21.26	540	71.9	9178	2797	71
20.94	532	70.9	9545	2909	70
20.63	524	69.9	9917	3023	69
20.35	517	68.9	10293	3137	68
20.04	509	67.9	10673	3253	67
19.76	502	66.9	11058	3371	66

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Oxygen Solubility Table Solubility of Oxygen (mg/L) in Water Exposed to Water-Saturated Air at 760 mm Hg Pressure

Temp °C	Chlorinity: 0 Salinity: 0	5.0 ppt 9.0 ppt	10.0 ppt 18.1 ppt	15.0 ppt 27.1 ppt	20.0 ppt 36.1 ppt	25.0 ppt 45.2 ppt
0.0	14.62	13.73	12.89	12.10	11.36	10.66
1.0	14.22	13.36	12.55	11.78	11.07	10.39
2.0	13.83	13.00	12.22	11.48	10.79	10.14
3.0	13.46	12.66	11.91	11.20	10.53	9.90
4.0	13.11	12.45	11.61	10.92	10.27	9.66
5.0	12.77	12.02	11.32	10.66	10.03	9.44
6.0	12.45	11.73	11.05	10.40	9.80	9.23
7.0	12.14	11.44	10.78	10.16	9.58	9.02
8.0	11.84	11.17	10.53	9.93	9.36	8.83
9.0	11.56	10.91	10.29	9.71	9.16	8.64
10.0	11.29	10.66	10.06	9.49	8.96	8.45
11.0	11.03	10.41	9.84	9.29	8.77	8.28
12.0	10.78	10.18	9.62	9.09	8.59	8.11
13.0	10.54	9.96	9.42	8.90	8.41	7.95
14.0	10.31	9.75	9.22	8.72	8.24	7.79
15.0	10.08	9.54	9.03	8.54	8.08	7.64
16.0	9.87	9.34	8.84	8.37	7.92	7.50
17.0	9.67	9.15	8.67	8.21	7.77	7.36
18.0	9.47	8.97	8.50	8.05	7.62	7.22
19.0	9.28	8.79	8.33	7.90	7.48	7.09
20.0	9.09	8.62	8.17	7.75	7.35	6.96
21.0	8.92	8.46	8.02	7.61	7.21	6.84
22.0	8.74	8.30	7.87	7.47	7.09	6.72
23.0	8.58	8.14	7.73	7.34	6.96	6.61
24.0	8.42	7.99	7.59	7.21	6.84	6.50
25.0	8.26	7.85	7.46	7.08	6.72	6.39
26.0	8.11	7.71	7.33	6.96	6.62	6.28
27.0	7.97	7.58	7.20	6.85	6.51	6.18
28.0	7.83	7.44	7.08	6.73	6.40	6.09
29.0	7.69	7.32	6.96	6.62	6.30	5.99
30.0	7.56	7.19	6.85	6.51	6.20	5.90
31.0	7.43	7.07	6.73	6.41	6.10	5.81
32.0	7.31	6.96	6.62	6.31	6.01	5.72
33.0	7.18	6.84	6.52	6.21	5.91	5.63
34.0	7.07	6.73	6.42	6.11	5.28	5.55
35.0	6.95	6.62	6.31	6.02	5.73	5.46
36.0	6.84	3.52	6.22	5.93	5.65	5.38
37.0	6.73	6.42	6.12	5.84	5.56	5.31
38.0	6.62	6.32	6.03	5.75	5.48	5.23
39.0	6.52	6.22	5.98	5.66	5.40	5.15
40.0	6.41	6.12	5.84	5.58	5.32	5.08
41.0	6.31	6.03	5.75	5.49	5.24	5.01
42.0	6.21	5.93	5.67	5.41	5.17	4.93
43.0	6.12	5.84	5.58	5.33	5.09	4.86
44.0	6.02	5.75	5.50	5.25	5.02	4.79
45.0	5.93	5.67	5.41	5.17	4.94	4.72

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+1.937.767.7241
info@ysi.com
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SOP No. S-3

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
DECONTAMINATION OF FIELD EQUIPMENT

STANDARD OPERATING PROCEDURE
DECONTAMINATION OF FIELD EQUIPMENT

1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the methods to be used for the decontamination of all field equipment which becomes potentially contaminated during a sample collection task. The equipment may include split-spoons, bailers, trowels, shovels, hand-augers, screens, or any other type of equipment used during field activities.

Decontamination is performed as a quality assurance measure and a safety precaution. It prevents cross-contamination between samples and also helps to maintain a clean working environment for the safety of all field personnel.

Decontamination is mainly achieved by rinsing with liquids which may include: soap and/or detergent solutions, tap-water, deionized water, acid solutions, and methanol. Equipment will be allowed to air dry after being cleaned or may be wiped dry with clean cloth or paper towels if immediate re-use is needed. The frequency of equipment use dictates that most decontamination be accomplished at each sampling site, between collection points. Waste products produced by the decontamination procedures, such as waste liquids, solids, rags, gloves, etc. must be collected and disposed of properly. All decontamination materials and wastes should be stored in a central location so as to maintain control over the quantity of materials used and/or produced throughout the study.

2.0 RESPONSIBILITIES

It is the primary responsibility of the project Field Operations Leader (FOL) and field samplers to assure that the proper decontamination procedures are followed and that all waste materials produced by decontamination are properly stored and disposed.

It is the responsibility of the project safety officer to draft and enforce safety measures which provide the best protection for all persons involved directly with sampling and/or decontamination.

It is the responsibility of any subcontractors (i.e., drilling contractors) to follow the proper designated decontamination procedures that are stated in their contracts and outlined in the Site Safety and Health Plan (SSHP).

It is the responsibility of all personnel involved with sample collection or decontamination to maintain a clean working environment and to ensure that any contaminants are not negligently introduced to the environment.

3.0 EQUIPMENT AND MATERIALS

3.1 Cleaning Liquids

Cleaning liquids may include tap (potable) water, deionized water, and soap and/or detergent solutions, nitric acid solutions, and methanol. For the site, only deionized water and Liquinox®

will be used unless specified in the Sampling and Analysis Plan (SAP) for a specific sampling location.

3.2 Personal Safety Gear

Personal protective equipment (PPE) will be defined in the SSHP, and will include at a minimum; disposable chemical resistant gloves and safety glasses or goggles.

3.3 Materials and Cleaning Containers

Materials may include paper towels, shop rags, various brushes, plastic or stainless steel buckets, plastic tubs, stainless steel manual sprayers, and spray bottles.

3.4 Waste Storage Containers

Waste storage containers may include metal or polyethylene drums, boxes, plastic bags, or buckets with lids.

4.0 PROCEDURES

4.1 General Approach

All equipment that comes in contact with the media that is sampled should be included in the decontamination process.

The standard procedures listed in the following section can be considered the procedure for full field decontamination. If different or more elaborate procedures are required for a specific task, they will be spelled out in the SAP. Such variations in decontamination may include following all, just part, or an expanded scope of the decontamination procedure stated herein.

4.2 Soil Sampling Equipment

1. Remove any solid particles from the equipment or material by brushing and then rinsing with clean water. This initial step is performed to remove gross contamination.
2. Wash equipment with a soap or detergent solution and brush.
3. Rinse with de-ionized water.
4. Repeat entire procedure or any parts of the procedure if necessary.
5. If sampling equipment is to be used immediately at another location, wrap the equipment in aluminum foil and store in a safe place.

4.3 Submersible Pump Decontamination Procedures

This procedure will be used to decontaminate submersible pumps (if used) and pump tubing between groundwater sample collection points and at the end of each day of use. For wells where dedicated tubing is being used, no decontamination of the tubing is needed. The dedicated tubing will be placed back into the monitoring well and only the pump will be decontaminated as described in the following subsections.

The following materials will be used:

- plastic-Nalgene or PVC upright cylinder
- 5-10 gallon plastic water storage containers

- Deionized water
- Stainless steel spray bottle
- Paper towels

Procedure:

1. During decontamination the submersible pump will be placed on a clean surface (sheet of plastic) or held away from the ground.
2. Clean the upright plastic-nalgene/PVC cylinder as described above in Section 4.2.
3. Decontaminate the outer surface of the submersible pump and the entire tubing using a potable water rinse followed by a deionized water rinse.
4. Place the submersible pump upright in the cylinder and fill the cylinder with potable water.
5. Continue pumping until the water in the cylinder is pumped down and air is drawn through the pump. If tubing is being decontaminated, continue pumping water through the pump until the tubing is full and overflowing. Continue pumping a volume of water that is twice the volume needed to fill the tubing and run the pump to dryness. At this time air pockets will be observed in the discharge line. Shut off the pump immediately.
6. Using the water remaining in the cylinder, rinse the sealed portion of the power cord and discharge tube by pouring the water carefully over the coiled lines.
7. Repeat steps 4 through 6 using deionized water. Pump or drain all the remaining water from the tubing.
8. When reaching the next monitoring well place the pump in the well casing and wipe dry both the power and discharge lines with a clean paper towel as the pump is lowered.

5.0 QUALITY CONTROL/QUALITY ASSURANCE

5.1 Quality Control

Quality control for the decontamination of field equipment involves the collection of field quality control samples including equipment blank samples. Frequency of collection of QC samples will be specified in the SAP.

5.2 Equipment Blank Procedures

Equipment blanks will be collected after decontamination of field equipment and will be specific to the type of sample equipment used.

Procedure:

1. After decontamination, laboratory provided deionized water will be poured through the piece of sampling equipment that comes into contact with the field sample.
2. The rinse water will be collected in the appropriate sample containers (see SAP).
3. The sample containers will be labeled with the appropriate nomenclature to designate the blank (see SAP).

4. Information regarding the equipment blank will be recorded in the field logbook and will include at a minimum: date, time, type of equipment, and sample dates associated with blank.
5. The equipment blank will then be packaged and shipped with the next sample set sent to the analytical laboratory.

6.0 REFERENCES

U.S. Environmental Protection Agency (USEPA), January, 1986. "Decontamination Techniques for Mobile Response Equipment Used at Waste Sites (State-of-the-Art Survey)." EPA/600/52-85/105.

USEPA, March, 1985. "Guide for Decontaminating Buildings, Structures, and Equipment at Superfund Sites." EPA/600/2 85/028.

SOP No. S-4

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
SAMPLE PACKAGING AND SHIPMENT

STANDARD OPERATING PROCEDURE

SAMPLE PACKAGING AND SHIPMENT

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes methodologies for shipping samples collected during environmental field investigation/remediation activities. This SOP applies to all environmental samples including drinking water, groundwater, surface water samples, soil, and sediment samples, and treatment plant samples.

2.0 DEFINITIONS

Shipper's Declaration – A paper document describing the contents of a shipment.

3.0 HEALTH AND SAFETY WARNINGS

Shippers of dangerous goods should take all precautions to eliminate any hazards associated with the goods being shipped. The shipper should consult the most-recent version of the International Air Transportation Association (IATA) regulations regarding shipment of dangerous goods.

4.0 PERSONNEL QUALIFICATIONS

Any person designated as a shipper of dangerous goods shall be trained in the U.S. Department of Transportation Hazardous Materials Regulations, which must be renewed every two years.

Shipment of environmental samples does not require specialized training; however, a familiarity with the regulations and the materials being shipped is considered beneficial.

5.0 EQUIPMENT AND SUPPLIES

Consult the most-recent version of the IATA regulations for a listing of proper shipping materials.

- Cooler -Samples -Labels -Ink pen
- Packing materials (bubble wrap) to prevent breakage, absorb leakage, and insulate samples.
- Polyethylene zip-type baggies large enough to contain the largest sample bottles.
- Custody seals if shipped through Federal Express (FEDEX) or similar shipping vendor.
- Large plastic trash bag to act as containment for the packing materials.

6.0 PROCEDURES

1. Be certain that all containers are sufficiently tight, preserved, and labeled correctly. Sediment samples should be allowed to settle for a minimum of 2 hours prior to shipping to the laboratory. The sample manger should look closely at all sediment samples to see if a clear water layer forms above the sediment. Any water layer should be decanted from the sample jar prior to shipping to the laboratory.
2. Clean the exterior of each sample container such that no gross contamination remains.
3. Complete the Chain of Custody (COC) as described SOP S-5. When the COC form is completed, verify that bottle labels, analytical fractions, and bottle numbers match what is written on the COC form.
4. Wrap sample containers in bubble wrap. Zip-type plastic baggies may be used as additional containment.
5. Line the cooler with the trash bag and add a layer of packing material. If the cooler has a drain, close and seal to prevent leakage of water from melting ice.
6. Place sample containers into the cooler, and pack them sufficiently to prevent them from shifting during shipment.
7. Place ice-filled zip-type bags on samples such that all samples are contacted by the ice. Place sufficient ice to retain the sample temperature between 2 and 6 degrees C. Place a temperature blank in with the samples.
8. Fill the remaining space in the cooler with packing material and close and secure the top of the trash bag.
9. On the chain of custody, sign in the relinquished by box and add in the subsequent received by box the name of the courier/carrier and the air bill No. (if applicable).
10. Place the COC into a plastic bag and tape it to the inside top of the cooler.
11. Close the cooler and tape the cooler shut with strapping tape or similar high-strength shipping tape.
12. If more than one cooler is being shipped under the same COC, copies of the COC should be placed into each additional cooler in the same manner as the original COC.
13. If shipped through FEDEX or other shipping vendor, apply custody seals to the cooler such that the seals must be broken in order to open the cooler.
14. Apply "UP Arrows" in the appropriate direction on at least opposing sides of the cooler exterior or indicate on top "this side up.
15. Add the appropriate shipping address labels to the cooler along with a return address to the cooler. If more than one cooler is being shipped, add "one of ____" to the label so that the recipient is aware that more than one cooler should be received.

7.0 DATA AND RECORDS MANAGEMENT

A copy of the COC shall be retained by the shipper until the completed laboratory data package is received. In addition, a copy of the air bill shall also be retained for validation/custody purposes and also for payment.

8.0 REFERENCES

Wood Environment & Infrastructure Solutions Inc., Standard Operating Procedure for Chain of Custody S-5 Code of Federal Regulations 40 CFR Part 261.4(d) Samples. Dangerous Goods Regulations, IATA, Most-Current Version.

SOP No. S-5

Wood Environment & Infrastructures Solutions, Inc.

STANDARD OPERATING PROCEDURE
SAMPLE CHAIN OF CUSTODY PROCEDURE

STANDARD OPERATING PROCEDURE

SAMPLE CHAIN OF CUSTODY PROCEDURE

1.0 INTRODUCTION

This standard operating procedure (SOP) describes chain of custody procedures to be followed whenever collecting environmental samples. This SOP is referenced in all SOPs for environmental sample collection.

2.0 MATERIALS

2.1 Documentation

- Work Plan
- Field Data Records (FDR)
- Chain-of-custody forms
- Sample labels
- Field logbook
- Permanent marker
- Lab contact information
- Chain-of-Custody Form

3.0 PREPARATION

Review Work Plan to identify samples to be collected, analyses to be performed, laboratory performing the analyses, and any other project specific-objectives of the sampling program. Review sample collection SOPs for media being sampled.

4.0 SAMPLE LABELING

Enter in the log book and label each sample container with the following information:

a) Charleston AFS project number;

b) Date and time of collection;

c) Sample location;

d) Sample number;

e) Analysis to be performed;

f) Sampler's initials;

g) Preservative

If using field sample tracking system labels will be generated and printed by the field sample coordinator.

5.0 CHAIN OF CUSTODY

5.1 Definition

EPA provides the following definition of chain-of-custody (COC):

"A sample is considered to be in your custody if any of the following criteria are met:

- The sample is in your possession or is in your view after being in your possession;
- The sample was in your possession and then locked up or sealed to prevent tampering; or
- You have placed the sample in a secured area.

5.2 Purpose

The chain-of-custody form is functionally similar to a packing slip that accompanies a shipment of goods. The chain-of-custody form includes a chain-of-custody record located at the bottom of the form. The form is used as physical evidence of sample custody. EPA guidelines specify that official custody of samples must be maintained and documented from the time of collection until the time the samples are introduced as evidence in the event of litigation. The sampler is responsible for the care and custody of the sample until sample shipment.

5.3 Documentation

After samples are collected and labeled, fill out the chain-of-custody form. The sampler becomes the initial sample custodian.

Chain-of-custody forms must be completed for every shipment of samples to an analytical laboratory.

Use indelible ink only, no pencil (a ball point pen is best). Make corrections by drawing a line through and initialing and dating the error, then enter the correct information. Erasures are not allowed.

A separate chain-of-custody form must accompany each cooler for each shipment. Place the original COC form in a zipper-type plastic bag in the cooler with the samples. The chain-of-custody forms must address all samples in that sample shipment. If multiple coolers are shipped a copy of the COC should accompany each cooler. This practice maintains the chain-of-custody for all samples in case of mis-shipment.

5.4 Transfer of Custody

When transferring the possession of samples, the individuals relinquishing and receiving custody will sign, date, and note the time on the record. Persons receiving the custody of a sample group are responsible for confirming the accuracy of the COC with regard to the number and type of sample containers for which they are accepting responsibility.

When samples are to be shipped to an analytical facility by commercial delivery service, the samples will be relinquished to the courier in sealed containers, and, if practicable, the shipment number will be noted on the COC form. When samples are transferred by commercial delivery service, a copy of the shipping documentation will serve as the COC record for the delivery service's role in the chain of custody.

The sample custodian relinquishing custody to a facility or agency will request the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this will be noted in the "Received by" space on the COC. When appropriate, the custody record will contain a statement that the samples were delivered to the designated location at the designated time.

6.0 REFERENCES

ASTM D4840-99, 1999; "Standard Guide for Sample Chain-of-Custody Procedures"; December 10, 1999; published January 2000

U.S. EPA Region 4; 2013; "Sample and Evidence Management", SESDPROC-005-R2, January 29, 2013; Revision 2

SOP No. S-6

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
USE OF FIELD LOGBOOKS

STANDARD OPERATING PROCEDURE

USE OF FIELD LOGBOOKS

1.0 SCOPE AND APPLICABILITY

The use of a Site Logbook and Field Logbook provides a daily record of significant events, observations, and measurements during field investigations. A site logbook is the master log for recording activities during an investigation. Field logbooks provide data and observations which will enable field personnel to reconstruct field project events. Sufficient data and observations should be logged in the field logbook to enable reconstruction of field events and to provide sufficient evidence in the event of legal proceedings.

2.0 RESPONSIBILITIES

It is the responsibility of the Field Operations Lead (FOL) to maintain centralized daily log book records of all significant field events, observations, and measurements during field investigations. All members of the field team are responsible for maintaining complete records of their actions, observations, etc. in their log books and providing this information to the team leader at the end of each day. If observations and measurements are taken in an area where the field log book may become contaminated or if the field personnel are spread over a large area, separate waterproof bound and numbered field log books may be maintained. Logbook entries should be signed and dated at the completion of each task or at the end of each day. Individual field log books are retained by the field team members until the logbook is filled or the completion of the project, at which time, possession of the log books is transferred to the FOL or project manager.

Errant field entries shall have a single line drawn through them and the correct data entered above it. All corrections shall be initialed and dated by the appropriate field personnel. Individual pages should never be removed from bound logbooks.

3.0 EQUIPMENT DESCRIPTIONS

A waterproof, bound field notebook and indelible ink pen are the standard field equipment.

4.0 PROCEDURES

The title page of each logbook will contain the following:

- The logbook number
- Project name and project number
- Site name and address
- Logbook start date

The site logbook and field logbooks provide a daily hand written account of all field activities. All entries are made in permanent black or blue ink, and corrections are made with a single line with the author initials and date. Each page of the logbook will be dated and signed by the person completing the log. Partially completed pages will have a line drawn through the unused portion at the end of each day.

Site Logbook

The site logbook is a record of all major tasks completed for each day or operation. Entries are made each day. The FOL responsible for on-site field operations will complete the site logbook. At a minimum the site logbook will contain the following information:

- A list of all field logbooks created for the project;
- Names and titles of all project related personnel present at the site during each day of operation;
- A brief summary of all activities completed for each day of operation;
- A listing of any changes made to established SI/RI program procedures; and,
- A summary of any problems encountered during the day including a description of corrective actions and impacts on the project.

Field Logbook

Field logbooks are daily records of field task activities that are entered in real time by the on-site field technicians and scientists. The following information is entered into the field logbooks:

- The date and time of each entry. The daily log should begin with weather conditions and the names and organizations of personnel performing the documented task;
- A summary of important tasks or subtasks completed during the day;
- A description of any field tests completed in association with the daily task;
- A description of any samples collected including documentation of any quality control samples that were prepared (rinse blanks, duplicates, matrix spikes, split samples);
- Documentation of equipment maintenance and decontamination activities; and,

- A summary of any problems encountered during the day including a description of corrective actions and impacts on the daily task.

SOP No. S-7

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

PROCEDURE FOR DESCRIPTION AND IDENTIFICATION OF SOILS

STANDARD OPERATING PROCEDURE

PROCEDURE FOR DESCRIPTION AND IDENTIFICATION OF SOILS

1.0 SCOPE AND APPLICABILITY

The appearance and textural properties of soil samples will be described using the Unified Soil Classification System (USCS). The USCS uses grain size to divide soils into different soil classes, coarse grained vs. fine grained. The system then further describes the soils based on the mix of coarse materials such as sand and gravel or the relative plasticity of the fine grained materials such as silt and clay.

Soil type identifications and descriptions will be recorded by field samplers during field investigation activities. Soil types will determined when completing explorations (monitoring well installations, soil borings, and surface soil sampling) and other activities where descriptions of soils are needed to characterize site location conditions. These field descriptions may be supplemented with laboratory data on grain size distributions analyses to characterize soils.

2.0 EQUIPMENT AND SUPPLIES

- USCS Key
- 6 foot folding rule or other measuring tool
- PID
- Field Data Records
- Knife of spatula

3.0 PROCEDURE

Soil descriptions are made using the USCS Classifications and will include the following observations:

- Color
- Name
- Gradation
- Density
- Moisture
- Plasticity
- Structure
- geologic origin
- USCS classification designation.

A USCS key to soil descriptions and terms is included as Attachment 1. All sample descriptions will be recorded in a field log book and/or the Field Data Record for the media being sampled (see Sampling and Analysis Plan).

Attachment 1 USGC Classification Chart

wood.

KEY TO SOIL DESCRIPTIONS AND TERMS

UNIFIED SOIL CLASSIFICATION SYSTEM

MAJOR DIVISIONS			GROUP SYMBOLS	TYPICAL NAMES
COARSE-GRAINED SOILS (more than half of material is larger than No. 200 sieve size)	GRAVELS (more than half of coarse fraction is larger than No. 4 sieve size)	CLEAN GRAVELS (little or no fines)	GW	Well-graded gravels, gravel-sand mixtures, little or no fines.
			GP	Poorly-graded gravels, gravel sand mixtures, little or no fines.
		GRAVEL WITH FINES (Appreciable amount of fines)	GM	Silty gravels, gravel-sand-silt mixtures.
			GC	Clayey gravels, gravel-sand-clay mixtures.
	SANDS (more than half of coarse fraction is smaller than No. 4 sieve size)	CLEAN SANDS (little or no fines)	SW	Well-graded sands, gravelly sands, little or no fines
			SP	Poorly-graded sands, gravelly sand, little or no fines.
		SANDS WITH FINES (Appreciable amount of fines)	SM	Silty sands, sand-silt mixtures
			SC	Clayey sands, sand-clay mixtures.
FINE-GRAINED SOILS (more than half of material is smaller than No. 200 sieve size)	SILTS AND CLAYS (liquid limit less than 50)		ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands, or clayey silts with slight plasticity.
			CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, clean clays.
			OL	Organic silts and organic silty clays of low plasticity.
	SILTS AND CLAYS (liquid limit greater than 50)		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
			CH	Inorganic clays of high plasticity, fat clays.
			OH	Organic clays of mediumto high plasticity, organic silts.
	HIGHLY ORGANIC SOILS		Pt	Peat and other highly organic soils.

Desired Soil Observations: (in this order)

Color (Munsell color chart)
Name (sand, silty sand, clay, etc., including portions - trace, little, etc.)
Gradation (well-graded, poorly-graded, uniform, etc.)
Density/Consistency (from above)
Moisture (dry, damp, moist, wet, saturated)
Plasticity (non-plastic, slightly plastic, moderately plastic, highly plastic)
Structure (layering, fractures, cracks, etc.)
Geologic Origin (till, marine clay, alluvium, etc.)
Unified Soil Classification Designation

wood.
511 Congress Street
Suite 200
Portland, ME 04101
(207) 775-5401

TERMS DESCRIBING DENSITY/CONSISTENCY

Coarse-grained soils (more than half of material is larger than No. 200 sieve): Includes (1) clean gravels; (2) silty or clayey gravels; and (3) silty, clayey or gravelly sands. Consistency is rated according to standard penetration resistance.

Descriptive Term (Modified Burmister System)	Portion of Total
trace	0% - 10%
little	10% - 20%
some	20% - 35%
adjective (e.g. sandy, clayey)	35% - 50%

Density of Cohesionless Soils	Standard Penetration Resistance N-Value (blows per foot)
Very loose	0 - 4
Loose	4 - 10
Medium Dense	10 - 30
Dense	30 - 50
Very Dense	> 50

Fine-grained soils (more than half of material is smaller than No. 200 sieve): Includes (1) inorganic and organic silts and clays; (2) gravelly, sandy or silty clays; and (3) clayey silts. Consistency is rated according to shear strength as indicated.

Consistency of Cohesive soils	SPT N-Value blows per foot	Approximate Undrained Shear Strength (psf)	Field Guidelines
Very Soft	0 - 2	0 - 250	Fist easily Penetrates
Soft	2 - 4	250 - 500	Thumb easily penetrates
Medium Stiff	4 - 8	500 - 1000	Thumb penetrates with moderate effort
Stiff	8 - 15	1000 - 2000	Indented by thumb with great effort
Very Stiff	15 - 30	2000 - 4000	Indented by thumbnail
Hard	>30	over 4000	Indented by thumbnail with difficulty

Rock Quality Designation (RQD):

$$RQD = \frac{\text{sum of the lengths of intact pieces of core* > 100 mm}}{\text{length of core advance}}$$

*Minimum NQ rock core (1.88 in. OD of core)

Quality Description	RQD
Very Poor	<25%
Poor	25% - 50%
Fair	50% - 75%
Good	75% - 90%
Excellent	>90%

Desired Rock Observations: (in this order)

Color (Munsell color chart)
Texture (aphanitic, fine-grained, etc.)
Lithology (igneous, sedimentary, metamorphic, etc.)
Hardness (very hard, hard, mod. hard, etc.)
Weathering (fresh, very slight, slight, moderate, mod. severe, severe, etc.)
Geologic discontinuities/jointing:
-dip (horiz - 0-5, low angle - 5-35, mod. dipping - 35-55, steep - 55-85, vertical - 85-90)
-spacing (very close - <5 cm, close - 5-30 cm, mod. close 30-100 cm, wide - 1-3 m, very wide >3 m)
-tightness (tight, open or healed)
-infilling (grain size, color, etc.)
Formation (Waterville, Ellsworth, Cape Elizabeth, etc.)
RQD and Rock Mass Description (very poor, poor, fair, etc.)
Recovery

Sample Container Labeling Requirements:

Site
PIN
Boring Number
Sample Number
Sample Depth
Blow Counts
Sample Recovery
Personnel Initials

SOP No. S-8

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

FIELD PRESERVATION OF VOA AND VPH SOIL SAMPLES

wood.

STANDARD OPERATING PROCEDURE

FIELD PRESERVATION OF VOA AND VPH SOIL SAMPLES

1.0 SCOPE AND APPLICATION

This purpose of this standard operating procedure (SOP) is to outline the steps associated with field preservation of soil samples for volatile organic analysis (VOA) in accordance with U.S. Environmental Protection Agency (USEPA) Method 5035 (USEPA, 1996). This SOP includes procedures applicable to off-site laboratory Method 8260 VOA and Massachusetts Department of Environmental Protection (MADEP) Method for the Determination of Volatile Petroleum Hydrocarbons (VPH) analyses specified in the Sampling and Analysis Plan (SAP). Specific steps and details are described for the primary tasks of sample container preparation, soil sample collection, sample container management and documentation, sample analysis, and target compound quantitation.

2.0 SUMMARY OF METHODS

2.1 Method 8260 VOA

For Method 8260 VOA, soil and sediment samples will be preserved in water (low concentration) and methanol (high concentration) at the time of sample collection. Soils will be obtained from sampling devises (i.e., hand augers, split spoons or other auger sample collection apparatus, Geoprobe cores) using plastic syringe samplers used to reduce exposure of samples to air. Approximately 5 grams of soil for low concentration volatile organic compounds (VOC) and 10 grams of soil for high concentration VOC vials will be immediately transferred to a vial containing a pre-measured amount of preservation fluid. Vials will be transported to the laboratory for analysis using procedures specified in the Quality Assurance Project Plan (QAPP).

- For low concentration VOCs, two vials will be collected at each location. Vials must be shipped to the laboratory each day or frozen within 48 hrs of collection. When freezing water preserved samples, vials should rest on their side to prevent glass from cracking during freezing.
- For VOCs, one high concentration methanol vial will be collected at each location.
- For locations selected for matrix spike analysis, the number of vials will be tripled (6 low and 3 high concentration vials).

2.2 MADEP VPH

For MADEP VPH analyses, it is mandatory to preserve soil and sediment samples in methanol. Soils will be obtained from sampling devises (i.e., hand augers, split spoons or other auger sample collection apparatus, Geoprobe cores) using Soil/sediment samples must be collected in a manner that minimizes sample handling, environmental exposure and/or aeration.

- The use of specially designed air-tight collection samplers or a 30-mL plastic syringe with the end sliced off is recommended. All soil/sediment must be removed from the glass threads of the vial to ensure an adequate seal. Samples must be cooled to 0-6ºC immediately after collection. Plastic syringe samplers used to reduce exposure of samples to air.
- Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.
- The desired ratio of methanol-to-soil/sediment is 1 mL methanol/1 gram soil/sediment, + 25%. The exact weight of the soil/sediment sample and volume of methanol must be known or ascertained by the laboratory when calculating and reporting soil/sediment concentration data. A recommended practice is for a laboratory to provide labeled, pre-weighed sampling vials with the measured volume of methanol clearly indicated to the field sampling technician. The laboratory "fill line" indicating the height of the methanol meniscus should be permanently marked on the side of the sampling container. After the soil/sediment sample is added to the methanol in the sampling container, the sample "fill line" indicating the height of the sample-displaced (increased) methanol level should also be marked by the field sampling technician. In all cases, the soil/sediment sample in the vial must be completely covered by methanol.
- Samples for VPH analysis should be collected in duplicate 60-mL or 40-mL VOC vials with Teflon-lined septa screw caps. An additional sample of the soil/sediment must also be obtained (without methanol) to allow for a determination of moisture content and VPH dry weight correction factors.
- A methanol trip blank should accompany each batch of soil/sediment samples.

3.0 EQUIPMENT AND SUPPLIES

- 40 milliliter (ml) glass VOA vial Teflon lined silicone septa lids filled with preservatives by the laboratory and pre-weighed.
- 20 ml plastic sampling syringe
- analytical balance capable of weighing to 0.1 gram
- utility knife
- stainless steel spatula
- vial storage cooler
- water resistant sample labels
- water proof marker

- Field Data Record (See SAP)

4.0 PROCEDURES

4.1 Sample Container Preparation

Sample containers used for the collection of off-site VOA and VPH samples will be prepared in advance at the off-site laboratory. Container preparation by the off-site laboratory will include attaching labels, adding preservation fluid, weighing sample containers, and recording all information necessary to document container preparation and to calculate sample weight and target analyte concentrations during subsequent sample analyses. Developing and implementing the exact procedures for container preparation will be the responsibility of the contract laboratory. The following requirements are provided to the contract laboratory for incorporation into the off-site laboratory procedures:

1. Containers will be prepared for each VOA or VPH sample.
2. The sample container will consist of a wide mouth glass vial appropriate for VOA soil samples. The container must contain a Teflon lined cap with an air tight silicone or phenolic septa.
3. A water resistant sample label will be attached to each container. Each sample container will be assigned a unique sample container tracking number that is marked on the container label with permanent waterproof ink. The label will have room for field samplers to record sample identification (ID), date sampled, time sampled, and initials.
4. Low concentration VOA vials will contain a stirring bar and 5 mL of water. High concentration VOA sample containers will be filled with 10 ml of purge and trap grade methanol. VPH vials will be filled with 10 mL of purge and trap grade methanol. The lot number of the methanol must be recorded. If possible, the laboratory will use a single methanol lot for the preparation of all VOA containers, or the laboratory should use as few lots as necessary for the program.
5. Sample container caps will be firmly capped to create an air-tight seal. Containers will be weighed and container weights will be recorded to the nearest 0.1 gram. No other tape or packaging material will be added to the containers. The laboratory will mark the approximate level of the methanol on the vial with a permanent marker. Containers will be stored in a designated location that does not contain other environmental samples or standards until shipment to the field. Containers will be shipped to the field office.
6. The laboratory will maintain container preparation records. Record keeping can be done using a bound notebook or preprinted forms. Records must contain all information necessary to document container preparation steps and calculate soil weights for each sample. These records will be submitted as laboratory notebook records with the analytical data deliverable packages. At a minimum the following information must be recorded:
    - preparation dates

- container tracking number for each container
- manufacturer and lot number of the containers
- methanol supplier and lot number
- pre-sampling weight of container and methanol (with cap and label on) recorded to the nearest 0.1 gram
- signature or initials of the individual preparing the containers
- additional fields for entering the post-sampling weight of container and the calculated weight of soil added to the container during sampling
- signature or initials of individual recording and calculating final weights

7. Containers will be stored at the field office in a dedicated area away from samples of sources of contamination. After sample collection and shipment to the laboratory, containers will be re-weighed by the off-site laboratory sample manager and the weights will be recorded into the container preparation records for use in calculating the actual soil weights for each sample.
8. **A separate sample vial filled with soil will be submitted for percent moisture determination in association with each soil sample that is collected from a saturated location (sediment) or from any location where only a VOC or VPH sample is collected.** The moisture sample will be collected using the same technique as the preserved sample. The laboratory will homogenize the sample (without decanting standing water) prior to collecting an aliquot for moisture determination.
9. Samples will be analyzed in accordance with methanol extraction purge and trap procedures specified in the analytical method specified in the SAP or QAPP. **The laboratory will shake VOC high concentration and VPH samples as described in the referenced analytical methods prior to taking a methanol aliquot for analysis.**

4.2 SAMPLE COLLECTION

Sample collection will be performed with a disposable plastic syringe. The appropriate volume of soil collected in the syringe will be estimated prior to sampling to collect the appropriate weight of soil specified in Section 2.0. Field personnel will make note of preservation fluid levels on the sample containers to ensure no significant loss had occurred. Field personnel are responsible for ensuring that sample containers remain on ice at all times. The specific steps and details for soil sample collection are outlined below:

1. Using a clean utility knife or other sharp knife carefully cut of the tapered end of the 20 ml plastic sampling syringes. Take care to remove the tapered portion without removing significant portions of the body or tube of the syringe. Sampling syringes are disposable and are not to be reused after collecting a sample.
2. Transport sample containers in cooler with bagged ice. Keep sample containers in individual zip lock bags.

3. Obtain photoionization detector (PID) readings from the sample surface.

4. Samples are collected by capturing a representative sample within the sampling syringe and transferring the soil to the VOA vial. **For low concentration VOCs, two vials will be collected at each location. For VOCs one high concentration methanol vial will be collected at each location. For VPH two methanol vials will be collected at each location (see method summary).** If samples are collected using split spoons or a Geoprobe sampler, samples will be collected from the soil core immediately upon opening the sampling devise. If samples are collected from hand augers samples will be collected from within the auger core. For surficial sediments or test pits, samples will be collected directly from the sampling location substrate. Push/advance the sampling syringe into the center of the sample core/location filling the soil sampling syringe to the target level volume. Pull the syringe plunger back further to apply suction on the soil sample which will help it to remain in the syringe during removal. Separate the syringe sample from the remaining soil. Remove the syringe. If the proper volume of soil is not present, repeat the procedure until the proper volume of soil has been collected. If necessary, use a stainless steel spatula to fill the syringe with the needed soil volume. If rocks are present in the sample it may be necessary to extrude the sample from the sleeve, select a portion of the core sample that is void of large rocks, and then advance the sampling syringe. If possible, the sample volume should consist of sand, silt or clay and contain very few rocks or pebbles.

   Note: If matrix spike/matrix spike duplicate (MS/MSD) samples are required, additional sample volume is necessary. Low Level Concentrations require 2 vials of DI water and 1 vial of Methanol. Methanol is used to screen the sample. These screening results are interpreted and the appropriate analysis is performed.

5. Remove a sample container from the cooler. Carefully extrude the soil sample from the syringe into the sample container. This task should be done slowly and carefully to insure that the preservation fluid does not splash from the sample container. **A second vial used for moisture determination will be collected for all soils collected below standing water or at any location where only VOC samples are collected. The samples will be collected using the same sample syringe and coring technique used for the actual field sample. The sample jars will be labeled "percent moisture determination for VOA" with the sample label also containing all other sample information including sample ID, date and time sampled, and sampler initials.** The laboratories will be instructed to homogenize the VOA percent moisture sample in the jar prior to removing an aliquot for moisture determination to simulate actual sample moisture added to the methanol vials.

6. Syringes should be discarded immediately after extruding sample from syringe; <u>do not reuse</u>. If split samples are collected, care must be taken to make the samples equally representative (i.e., collected from the same part of the soil core).

7. Replace container cap as soon as possible.

8. With permanent waterproof ink fill out the sample container label with the following information: date, time, location, depth of sample, sample ID code, sample type (i.e., regular, duplicate, matrix spike, matrix spike duplicate), and sampler initials. The approximate level of the methanol will be marked on the sample vial. Do not tape over the sample container label.
9. Make sure the sample container lid is screwed down tightly. If necessary wipe excess soil from the mouth of the container to get an air-tight seal. Place the sample container back into the zip lock bag. Place the container and bag into the cooler taking care that the sample container remains upright. Keep samples on ice until they are submitted to the sample manager.
10. Complete the appropriate Field Data Record (See SAP) and release the samples to the sample manager.

5.0 SAMPLE MANAGEMENT

Upon completion of sample collection, sample containers will be released to a sample manager. The specific steps and details for sample management are outlined below for samples submitted to the off-site laboratory.

5.1 Off-Site Laboratory Samples

Sample containers will be weighed by the off-site laboratory sample manager immediately upon receipt at the off-site laboratory. The sample manager will record the container identification number and post-sampling container weight on the chain of custody. A trip blank will accompany each shipment of samples to the off-site laboratory. The trip blank will consist of a sample container containing water (low concentration) and one containing methanol (high concentration) prepared by the off-site laboratory for the same analytical method as the field samples.

6.0 REFERENCES

U.S. Environmental Protection Agency (USEPA), December, 1996. "Test Methods for Evaluating Solid Waste"; Laboratory Manual Physical/Chemical Methods; Office of Solid Waste and Emergency Response; Washington, DC; SW-846; November 1986; Revision 4.

Massachusetts Department of Environmental Protection (MADEP), January 2017. "Method for the Determination of Volatile Petroleum Hydrocarbons (VPH) by Gas Chromatography/Mass Spectrometry"; Commonwealth of Massachusetts Bureau of Waste Site Cleanup; January, 2017; Revision 0.

SOP No. S-9

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
SOIL HEADSPACE SCREENING PROCEDURE

wood.

STANDARD OPERATING PROCEDURE

SOIL HEADSPACE SCREENING PROCEDURE

1.0 SCOPE AND APPLICABILITY

1.1 PURPOSE:

To screen soil sample headspaces for total ionizable volatile organic compounds (VOCs). This is a semi-quantitative method used to identify the presences, absence, and relative concentrations of VOCs in soil. Headspace screening is performed with a photoionization detector (PID). Screening may used to:

1. Segregate soil piles;
2. Identify soil samples for laboratory analyses; and
3. Provide a qualitative assessment of the presence of VOCs for use in contamination assessments.

2.0 SUMMARY OF METHOD

A sample of soil is placed in a plastic bag or sample jar and sealed. The sample is shaken and held to allow VOCs to fill the air space. The air is tested for VOCs using a hand held detector.

3.0 HEALTH AND SAFETY WARNINGS

The site safety and health plan (SSHP) should be reviewed to determine the level of personal protection is required for work at site locations.

4.0 INTERFERENCES AND LIMITATIONS

- To be detected, compounds must be present, volatile in the state of the soil sample being screened, and capable of being ionized by the PID in use.
- Screening results will vary based on sample temperature, compounds present, age of the sample, and the degree to which the sample has been agitated and crumbled.
- VOCs in soil gas or groundwater within the soil pores will produce positive screening results even for soil samples which may not "contain" VOCs.
- Water vapor may cause large-scale zero drift in the PID.

5.0 MATERIALS

5.1 Screening

- PID, including calibration kit and manual
- Sample jars or plastic bags

- Indelible marker
- Aluminum foil

5.2 Recording

- Field logbook
- Field Data Record if applicable (See SAP)

5.3 Health and Safety Equipment per Work Plan

6.0 PREPARATION

Calibrate PID per manufacturer's instructions and document calibration in the field book. Make note of calibration or spanning to non-standard specifications.

Refer to SOP S-1 for soil sample collection procedures.

7.0 PROCEDURES

Record and document background VOC readings in ambient air. If it is not feasible to screen samples in an area with a clean background, document the highest background reading.

Half fill a clean jar or Ziplock™ type plastic bag with soil. Quickly cover the jar with aluminum foil or close the plastic bag and label the container.

Vigorously shake the sample to disperse soil and wait for approximately 5 minutes. Record the ambient temperature at which screening is performed. If outside temperatures are below 50°F, try to warm the samples in a heated vehicle or building.

Shake the sample again after 5 minutes.

Insert the tip of the PID through the foil or into the plastic bag and record the highest meter response, typically after approximately 3 to 15 seconds.

After screening all samples, re-check background and record significant variations.

8.0 REPORTING

The PID has a reliable reporting limit of 1 part per million in air. Readings at or below the reporting limit should be reported as not detected (ND), below detection limit (BDL), or similar terminology.

9.0 DECONTAMINATION

- Brush loose soil from the PID, etc.
- Containerize and handle decontamination wastes and wastewater in accordance with the SAP and SSHP.

SOP N0. S-10

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

SPILT-SPOON/SPLIT-BARREL SUBSURFACE SOIL SAMPLE COLLECTION AND STANDARD PENETRATION TEST PROCEDURE

wood.

STANDARD OPERATING PROCEDURE

SPILT-SPOON/SPLIT-BARREL SUBSURFACE SOIL SAMPLE COLLECTION AND STANDARD PENETRATION TEST PROCEDURE

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) is applicable to the collection of representative subsurface soil samples utilizing a split-spoon or split-barrel (split-spoon) sampler. In addition, this SOP provides the procedure for implementing the Standard Penetration Test (SPT). The results of an SPT yield a practical value of relative bearing capacity. The SOP is applicable for civil, environmental, and geotechnical investigations.

2.0 SUMMARY OF METHOD

The use of a split-spoon sampler provides either continuous or discrete collection of soil samples from the subsurface. In addition, the use of the sampler is an iatrical component of the SPT. The split spoon sampler is typically utilized in conjunction with conventional drilling techniques such as hollow stem auger or drive-and-wash drilling.

The assembled split-spoon sampler is hammer- or weight-driven into the soil such that a hollow core of the sampler is filled with soil and withdrawn from the boring. Once withdrawn from the boring, the split-spoon sampler is disassembled and the resulting sample is exposed for collection, field analysis(es), and/or classification.

The SPT is a test that results in a relative soil bearing capacity based upon the number of times the hammer or weight (of known mass and fall distance) drives the split-spoon two feet into the soil (aka. the "blow count").

Soil samples are collected for classification of lithology, field headspace screening (in the case of petroleum/chemical release investigations), and laboratory analysis (geotechnical and/or environmental in nature).

3.0 DEFINITIONS

Split-Spoon Sampler -open-ended cylindrical tool used to collect samples by driving or pushing them into the ground. Split-spoon samplers have inside diameters ranging from 3 to 6.3 cm (1-3/8 to 2-1/2 in.) and usually consist of five parts, similar to a continuous barrel sampler (Figure 1).

4.0 HEALTH AND SAFETY WARNINGS

This SOP does not address specific activity hazard analyses inherent with this procedure and cannot address all hazards associated with drilling activities. Therefore, health and safety details such as activity hazard analyses should be included and referenced in the Site Safety and Health Plan (SSHP). The following is a highlighted summary of health and safety issues associated with every split-spoon sampling event.

Prior to the advancement or **ANY** subsurface activity (e.g., drilling, excavation, etc.), calls to the appropriate underground utility clearance organizations such as DIGSAFE and municipal utility departments **MUST** be made, and utility clearance **MUST** be received. Records of this call should be available to field staff.

Split-spoon sampling involves the use of many moving parts, each of which may present a pinching hazard, overhead weights which may present crushing hazards, and sharp objects and hand tools which may present piercing, crushing, or repetitive motion hazards. In addition, driving the split-spoon with the weights/hammers may present a noise hazard.

Often, split-spoon sampling is involved in the investigation or remediation of a petroleum or hazardous material release. Particular chemical-specific hazards (which are detailed in the site health and safety plan) are present and specific safety parameters/controls are required.

5.0 CAUTIONS

If the potential for subsurface contamination exists at a location or is unexpectedly encountered during drilling, care should be used to prevent penetration of any geologic confining layer, which may impact the distribution of contamination. Thin confining layers may not be readily identified if noncontinuous sampling is utilized.

Split-spoon sampler damage may result from overdriving the sample, or if the sampler is driven into rock. Split-spoon sampling is generally not feasible in weathered rock.

Split-spoon sampling is generally more-difficult if a "running-sands" condition is encountered. Often, this condition can be accommodated using a drive and wash drilling method as opposed to a rotary auger method.

6.0 INTERFERENCES

If a split-spoon sample is collected from a cohesionless and unconsolidated aquifer, the groundwater in the formation may run out of the sampler once above the water table causing the fine-grained materials to "wash out" from the sample. If observed, this condition should be noted in field logs.

If soil samples are to be collected for volatile organic compounds (VOC) analysis (including field headspace screening), samples should be collected immediately upon separating the two halves of the sampler. SOPs for field headspace screening and preservation of soil samples for VOC analysis have been developed under separate cover. A delay in collection of VOC samples may result in an underestimation of VOC concentrations in the affected sample(s).

During environmental investigations, insufficient homogenization of the nonvolatile sample aliquots may result in a faulty estimation of contaminant concentrations.

7.0 PERSONNEL QUALIFICATIONS

Soil samplers utilizing this SOP should be familiar with the methods of drilling which coincide with the split-spoon sample collection technique.

Soil samplers utilizing this SOP should be proficient in visually-classifying soil samples via the Wood Environment & Infrastructure Solutions, Inc. SOP S-7 (Procedure for Description and Identification of Soils).

In the case of petroleum/chemical release investigations, personnel should be trained to the appropriate degree in hazardous waste site safety procedures as per OSHA regulations.

Any drill operators should be qualified to operate the drill and be licensed (if applicable) in the State in which the work is being performed. Drill operators should also be trained in hazardous waste site safety procedures as per OSHA regulations as needed.

8.0 EQUIPMENT AND SUPPLIES

The following list is not exhaustive but provides a listing of "at a minimum" equipment and supplies.

For all subsurface sampling events the following equipment is required:

- Drill equipment; Hollow Stem Auger rig, Barber drill rig, Air/Mud Rotary rig, etc.
- Split spoon samplers and drill rods
- Field logbook
- Stakes, pin flags, or other marker
- Folding engineer's rule
- Lumber crayon
- Labeled sample jars (for classification)
- Indelible marker
- Fiberglass engineer's tape measure
- Decontamination equipment
- Camera

For environmental investigations, the following equipment should be included:

- Stainless-steel bowl and mixing scoop/spoon
- Environmental sample containers
- Coolers
- Ice
- Chain of custody forms

- Appropriate personal protection equipment (PPE)

Additional optional equipment may include:

- Field penetrometer
- Field torvane
- Field gradational sieves

9.0 PROCEDURES

After the soil boring has been advanced to the desired sample depth and cuttings have been removed from the hole, the following procedure shall be followed for the collection of a representative subsurface soil sample. (Paraphrased from ASTM D 1586.)

Attach the split spoon to the drill rods and lower into the open bore hole. Do not allow the sampler to drop to the bottom of the boring.

Position the hammer/weight above the drill rods and attach the anvil to the top of the rod string.

Rest the entire weight of the rod string, anvil, and hammer on the sampler to determine the approximate amount of borehole slough is present. Remove if needed.

Mark the drill rods in 6-inch increments over two feet, such that the advance of the sampler is easily observed over each 6-inch increment.

Drive the sampler with blows from the 140-lb hammer, and record the number of blows per 6-inch increment over the 2-foot sample span. Continue driving the sampler until 1: a total of 100 blows have been applied, 2: there is no observable advance of the hammer over 10 consecutive blows, or 3: the sampler is advanced the entire 24-inch length of the sample span.

The sum of the 6”-12” and 12”-18” penetration is termed the “N-value” and should be recorded.

Bring the sampler to the surface using the drill rod and drill derrick. Open the sampler and record total recovery in tenths of a foot, and place a representative sample in a jar for classification (and in the case of petroleum/chemical release investigations also for headspace screening via Wood Environment & Infrastructure Solutions, Inc. SOP S-9 (Soil Headspace Screening Procedure).

If applicable, immediately upon opening and determining the appropriate sample collection depth, the VOC sample should be collected and directly transferred to the appropriate sample container and immediately preserved in accordance with Wood Environment & Infrastructure Solutions, Inc. SOP-8 (Field Preservation of VOA and VPH Soil Samples).

If environmental laboratory analytical samples are to be collected, the remaining split-spoon sample volume (after the VOC sample aliquot has been collected) shall be placed in a dedicated or decontaminated stainless-steel bowl and homogenized with a dedicated or decontaminated stainless-steel scoop/spoon for approximately 30 seconds to ensure sample homogeneity. A sample of the homogenized soil will then be collected and transferred to the appropriate soil

sample containers. The number, type, and size of the individual sample containers will vary depending on analysis to be performed and should be detailed in the approved site sampling and analysis plan or similar approved planning document.

If no environmental laboratory analytical samples are to be collected, the remaining split-spoon sample volume should be discarded or disposed of in an appropriate fashion with soil boring cuttings generated during the advancement of that specific boring.

Upon completion of sample collection, all sampling equipment should then be decontaminated in accordance with the approved sampling and analysis plan or other similar approved planning document.

All Investigation Derived Waste (IDW) shall be handled and disposed of in accordance with an approved sampling and analysis plan or other similar approved planning document.

10.0 DATA AND RECORDS MANAGEMENT

A record of conversation relative to utility clearance will be included in the project file.

A record of the visual classification of each sample will be retained in the project file in either field-logbook for or on a dedicated soil boring log.

If applicable, the sample classification jar should be retained until such time as it may be discarded. This may vary with contract/project requirements.

A photograph of a typical sample setup should be included in the project file as documentation that this SOP was followed adequately.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

Quality control and quality assurance parameters for sample collection vary from project to project, therefore, no procedures are set forth in this SOP. It should be noted that project sampling and analysis plans, quality control plan, or other similar planning document typically provide detailed information regarding QA/QC sample collection frequencies/schedules, and also decontamination procedures and verification sample collection, in particular as it applies to a petroleum/chemical release investigation.

12.0 REFERENCES

References cited as applicable to this SOP:

ASTM Designation D 1586-84 (Reapproved in 1992); Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

ASTM Designation D 2487-93; Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)

U.S. Environmental Protection Agency EPA/240/B-01/004; Guidance for Preparing Standard Operating Procedures (SOPs), March 2001

Cross-Referenced SOPs:

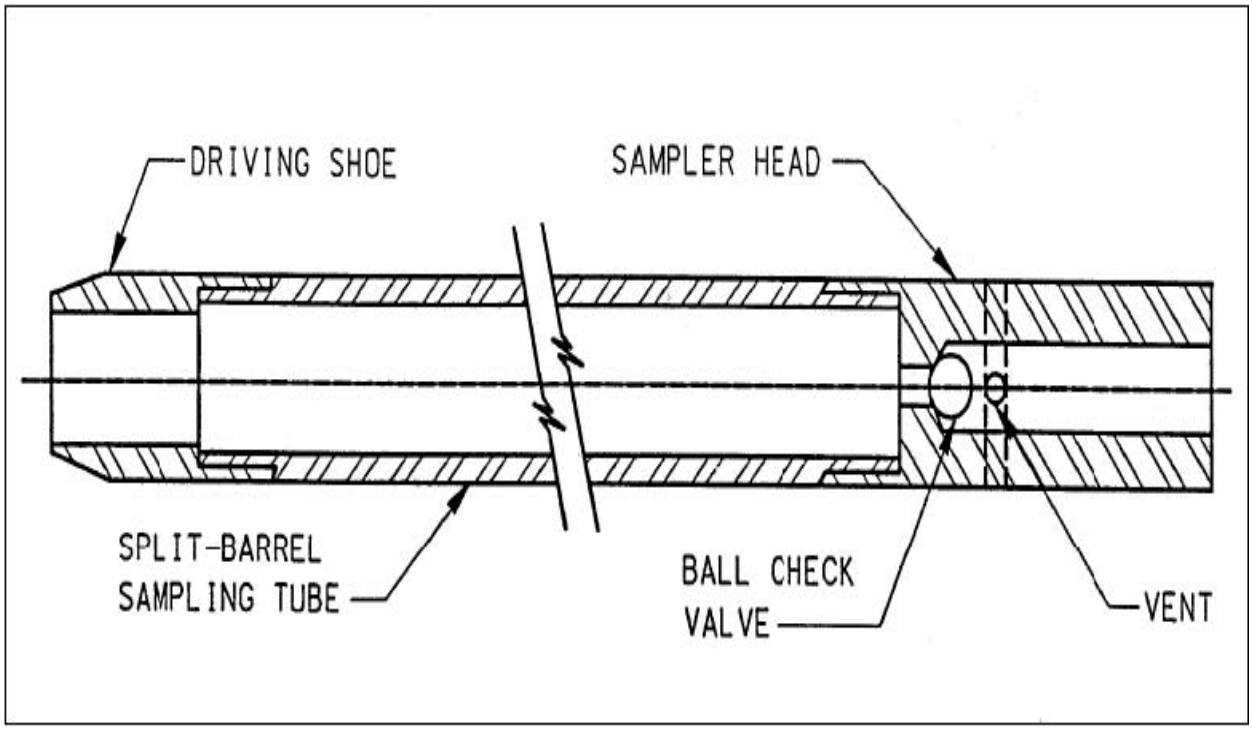
Wood Engineering & Infrastructure Solutions Inc., SOP No. S-7 for Description and Identification of Soils

Wood Engineering & Infrastructure Solutions Inc., SOP No. S-8 for Field Preservation of VOA and VPH Soil Samples

Wood Engineering & Infrastructure Solutions Inc., SOP No. S-9 for Soil Sample Headspace Screening for VOCs

13.0 FIGURES AND ATTACHMENTS

Figure 1. Schematic of a Split Spoon Sampler



SOP No. S-11

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
GEOPROBE DIRECT PUSH SAMPLING

wood.

STANDARD OPERATING PROCEDURE

GEOPROBE DIRECT PUSH SAMPLING

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes methodologies using a GeoProbe® direct-push sampling system that may be used to conduct soil, groundwater, or soil vapor sampling surveys. This technology can be used to collect samples for off-site laboratory analysis or provide screening information that can be used to optimize the future location of soil borings and monitoring well installations and to assess contamination in the vadose zone and saturated overburden. The project objectives and data quality objectives (DQOs) for the GeoProbe® sampling will be described in the sampling and analysis plan (SAP).

The direct-push explorations shall be completed by a qualified direct-push subcontractor and directed by a qualified field person.

2.0 SUMMARY OF METHOD

Direct-Push drilling technique consist of a hydraulic ram unit, usually mounted on a small vehicle (ATV, cargo van, or pick-up truck) that advances small diameter drill rods to obtain overburden soil or groundwater samples or install piezometers. The Geoprobe® pushes and/or hammers rods and probe tips into the subsurface for sample collection. Advantages in environmental investigations include low cost, maneuverability and access to irregular terrain, minimization of investigation derived wastes. Disadvantages include depth limitations and small sample volumes.

The direct push device may employ either dual tube methodology which allows the collection of subsurface soil samples through an outer casing that is set to maintain the integrity of the boring or single-rod method that collects soil into a sleeve liner within the lead rod.

In the dual-tube method borings are advanced by simultaneously driving an outer stainless steel casing and inner Lexan® tube into the ground. Upon reaching the desired penetration depth, the inner Lexan® tube is extracted to collect the discrete subsurface soil samples, leaving the outer casing in place. To sample the next interval of soil, a new length of Lexan® tubing is then inserted into the outer casing (already in the ground) attached to a length of drive pipe, and another length of outer casing is attached to the top of the outer casing that is already in the ground.

In the single-rod method, ¾-inch diameter rods are advanced in 4-ft sections. The lead section is fitted with an inner acetate sleeve. When the top of the desired sampling interval is reached, a tool is used to unlock the drive point and the rod is driven ahead to obtain the soil sample. The entire drill rod is retrieved and the liner removed for characterization. The process is then repeated to collect the next desired sample.

This process may be modified to collect groundwater samples or soil gas samples as described

in the groundwater and soil vapor procedures.

As with any heavy equipment, caution should be taken to minimize the potential for injuries such as crushing and pinching. Additionally, the drill rig has overhead hazards and in most cases noise hazards which should also be considered. Before any drilling is completed, the rig should be set level and the operator should inspect the location to verify that the unit is stable and secure enough to operate.

All personnel shall be familiar with the location of the rig's emergency kill switch.

All non-essential personnel should be kept clear of exclusion zone or from an area surrounding the rig.

Due to the potential of noise, ear protection is required. If needed, hand signals should be developed for communication between engineer and the driller.

3.0 PERSONNEL QUALIFICATIONS

The GeoProbe® operator should be familiar with the rig operations to adequately perform the sample collection task. The rig geologist/engineer should be sufficiently skilled in the drilling method and also be proficient in the classification of soils.

4.0 EQUIPMENT AND SUPPLIES

The following materials will be available, as required, during the subsurface soil sampling:

- Health and safety equipment;
- Direct push sampling equipment;
- decontamination equipment as specified in the QAPP;
- Stainless steel trowels or spatulas;
- Aluminum Foil;
- Paper Towels;
- Measuring device;
- Appropriate sample containers and Field Data Records (see Sampling and Analysis Plan [SAP])
- PID;
- Acetate field knife (if liner sleeves are used to collect the soil samples);
- Field notebook.
- Appropriate decontamination equipment (steam cleaner, materials for a decon pad, etc.) as necessary
- Drums for IDW containment as specified in the work plans
- PPE and monitoring equipment as specified in the Site Safety and Health Plan (SSHP)

- Piezometer construction materials if specified in the SAP.

7.0 PROCEDURES

Procedures are presented for soil sampling, groundwater sampling, and soil vapor sampling. Only procedures and media sampling specified in the project SAP will be completed. The operator should consult the SAP and field operation leader prior to completing investigation work.

7.1 Geoprobe® Soil Sampling

The following procedures will be employed to collect subsurface soil samples:

1. Identify sample locations from the Work and note the locations in field notebook by obtaining ties to physical features.
2. Obtain and wear the appropriate PPE.
3. Set up an equipment cleaning station, and decontaminate equipment as described in the SAP. Use new, clean materials when decontamination is not appropriate (e.g., disposable gloves and dedicated drive points). Document the decontamination procedure in the field notebook.
4. Assemble the appropriate direct-push sampling apparatus or other direct push tool. Soil samples will be collected using a three or four-foot long 1-to-2 inch diameter core sampler. The SAP will determine if a dual tube split-spoon system or a single rod acrylic liner method will be used for the collection of subsurface soil samples.
5. Drive the sampling tools to the appropriate sampling zone and collect a sample base on the type of direct-push method being used. Open the sampler by unscrewing the cutting shoe and retrieve the rod containing the soil sample. Open the sampler and cut open the acetate liner if used. Screen for VOCs using the PID. Collect the needed soils for laboratory analysis per requirements of the SAP. Measure and describe the sample lithology on the Soil Boring Log Field Data Record (See SAP) using the USCS Procedures for Description and Identification of Soils SOP S-7 (See SAP).
6. Evaluate the sample for the presence of visible non-aqueous phase liquid (NAPL). Document samples interpreted to contain visible NAPL, and record observations in field notebook and boring log.
7. Decontaminate non-disposable equipment or tools that may have come into contact with subsurface soil in accordance with the SAP.
8. Discard all disposable equipment used during sampling activities in a designated location.
9. Record sample collection information in the field notebook or Soil Boring Field Data Record including sample location, depth, PID readings, and analytical fractions collected.
10. Identify the next sequential boring location, move to that location and return to step 2

Records of each exploration shall be made on a Soil Boring Log (See SAP) and in the field logbook. All cuttings or other waste will be containerized or disposed of in accordance with planning documents.

7.2 GeoProbe® Groundwater Sampling

A direct-push sampling system (e.g., Geoprobe® or equivalent) may be used to obtain discrete groundwater grab samples if specified in the SAP. The collection of groundwater grab samples via the direct-push method is dependent on sufficient saturated thickness of overburden soils and an adequate rate of inflow through the probe tip.

1. A direct-push system advances a steel probe assembly to the desired depth indicated in the SAP as described in Section 7.1.
2. Groundwater samples are collected by allowing formation water to flow into a slotted probe tip or wire rapped stainless steel screen. Water within the probe is purged and sampled from inside the rod assembly using small-diameter tubing and a low-flow rate sampling pump, or a small-diameter bailer. One tubing volume of water will be purged and one set of parameters including temperature, conductivity, pH, and turbidity will be collected before sampling. VOC samples will be collected at a low purge rate (approximately 100 milliliters per minute) to minimize potential volatilization.
3. Sequential (vertical profile) sampling may be performed by driving the probe assembly to a predetermined depth and collecting a sample. Following sample collection, an additional section of riser is connected, and the sampling device is driven to the next sampling interval, where another sample is collected. Non-dedicated pumps and tubing shall be decontaminated and dedicated tubing shall be discarded between sample collection intervals.
4. Groundwater sample collection data shall be recorded on the Geoprobe Groundwater Grab/Pore Water Sampling Field Data Record (See SAP) and in the field logbook.

7.3 Geoprobe® Soil Vapor Sampling

Soil vapor samples may be collected using a GeoProbe® sampling device to provide data on the presence of VOCs in the subsurface vadose zone. Field data and observations will be recorded on the Soil Vapor Sampling Record. Samples may be analyzed at an off-site laboratory or with an on-site instrument. The Geoprobe® rods will be pushed to the desired sampling depth (expected to be below the rain infiltration line, but above the water table fringe zone).

<u>Procedure for GeoProbe® Soil Vapor Sample Collection in Summa Canisters</u>

Soil vapor samples will be collected from the Geoprobe® points using either the Geoprobe® PRT system, or through open Geoprobe® rods.

1a. To sample through the open rods, the rods are pushed down to the target depth and

then pulled back slightly, allowing a disposable point to drop off the bottom and expose the bottom of the open (hollow) rods to the soil. The rods will be sealed with O-rings at the joints and have a 1/4-inch tubing attached to the top for vapor purging and sample collection.

1b. To sample with the Geoprobe® PRT system, a specialized point is attached to the end of the Geoprobe® rods. The PRT point is also exposed to the soil by allowing a disposable tip to drop off the bottom of the rods when the rods are backed out slightly. This PRT point allows a ¼-inch tubing to be threaded directly to the bottom of the rods, for a small discrete sample point. The tubing is run to the surface and connected directly to the sample collection device.

1. For both techniques the outside of the rods will be sealed at the ground surface with pre-hydrated bentonite. Approximately 1 liter of soil vapor, plus the volume of the tubing or rods, will be purged using a personal air monitoring pump before collecting samples. During the soil vapor purge, vapors will be screened with a PID.

2. Soil vapor samples will be collected with either 1.4-liter SUMMA®-type canisters with flow valves (set to approximately 20 minutes per sample), or with Tedlar bags (Tedlar bags may be filled using either a Vac-U-Chamber®, or with a syringe with a three way valve).

<u>SUMMA® canister sample collection</u>

- Place SUMMA® canister adjacent to the temporary sampling port.
- Record SUMMA® canister serial number on sampling summary form and COC.
- Record sample identification on canister identification tag, and record on sampling summary form and COC.
- Remove plastic cap canister fitting.
- Open and close canister valve.
- Record gauge pressure on sample summary form and COC. Gauge pressure must read >25 inches Mercury (Hg). Replace SUMMA® canister if gauge pressure reads <25 inches Hg.
- Connect canister to silastic tubing already connected to the subsurface probe.
- Open canister valve and in-line stainless steel valve to initiate sample collection.
- Record date and local time (20-minute basis) of valve opening on sampling summary form and COC.
- Take digital photograph of SUMMA® canister and surrounding area.
- Upon completion of 20 minute sample collection, record gauge pressure on sampling

form and COC.

- Record date and local time (20 minute basis) of valve closing on sampling form and COC.
- Close canister valve.
- Disconnect silastic tubing and recap pressure gauge.
- Remove SUMMA® canister from sample collection area.
- Remove temporary probe from hole. Fill hole with a quick drying hydraulic cement.

Tedlar bag sample collection using Vac-U-Chamber®

1. The sampling line will be connected to a Vac-U-Chamber® Tedlar bag sampling box containing a one liter Tedlar sample bag.
2. The external pump is then connected to the purge port and the soil vapor sampling probe will be purged for two minutes prior to sample collection.
3. After purging the system, the external pump is connected to the vacuum port and the Tedlar bag is allowed to inflate. Upon complete inflation of the Tedlar bag, as observed through the Vac-U-Chamber® viewing window, the Tedlar bag valve is closed and the sample is labeled with the unique sampling location identification code.
4. If duplicate samples are collected, the duplicate sample will be collected by inserting a tee connector in the sampling line and filling two Tedlar bags from one probe at the same time.

Tedlar bag sample collection using syringe with a three way valve

1. The sampling line will be connected to the bottom port of a three way valve system.
2. A 60 to 100 milliliter (ml) syringe is then connected to the top purge port. The sampling line valve and the purge port are opened and the syringe is filled.
3. The sampling line valve is then closed and the side port is opened. The syringe is this emptied and the side port is closed.
4. A one liter Tedlar sample bag is connected to the three way valve side port. The sampling line valve and the purge port are opened and the syringe is filled again. The sampling line valve is then closed and the side port is opened. The contents of the syringe are then purged into the Tedlar bag. This process is continued until the Tedlar bag has been filled.

8.0 QUALITY CONTROL

For soil vapor events, helium leak tests will be conducted on a subset of samples to ensure samples are representative of sub-surface conditions and not outdoor ambient air. Helium leak tests will be conducted by encapsulating the sample point (such as with a bucket sealed to the ground surface with bentonite), while allowing the tubing to be purged from outside the

encapsulated area. The encapsulated area will be filled with helium, but care will be taken not to pressurize the enclosure. The soil vapor sample port will be tested for helium breakthrough with a portable monitoring device (such as the Radio detection MGD-2002 Multi-Gas Locator) both before and after collection of the soil vapor sample. If greater than 10 percent of the tracer gas is detected in the screening sample, the sample point seal will be enhanced and the procedure repeated.

9.0 DATA AND RECORDS MANAGEMENT

The following records will be generated by the site geologist or field sampler:

- Field Logbook entry
- Soil Boring Log Record (See SAP)
- Geoprobe Groundwater Grab/Pore Water Sampling Record (See SAP)
- Soil Vapor Sampling Record (See SAP)

SOP No. S-12

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
CALIBRATION PROCEDURE FOR PID

STANDARD OPERATING PROCEDURE

CALIBRATION PROCEDURE FOR PID

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes methodologies for calibration of the hand held photoionization detector (PID) used for field monitoring during sampling events. This procedures is designed to be used in conjunction with the instrument manufacturer instructions provided with the instruments as they are shipped into the field.

2.0 SUMMARY OF METHOD

Calibration is completed by analyzing a clean, zero air sample and a sample of span gas (isobutylene) of know concentration. The instrument software is used to set the PID response.

3.0 HEALTH AND SAFETY WARNINGS

Care should be taken when handling gas cylinders.

4.0 INTERFERENCES

The PID will respond to a variety of compounds that may be present as background in the ambient air. Testing of air at background locations or up wind locations may be necessary to understand the baseline response at a given site location.

Compound identification is not possible.

5.0 EQUIPMENT AND SUPPLIES

- Photoionization detector
- Zero gas cylinder
- Span gas cylinder
- Field Instrument Calibration Record
- Field logbook

6.0 PROCEDURES

Procedures are specified in the instrument manufacturer's instructions which are shipped with the instruments in the field.

7.0 DATA AND RECORDS MANAGEMENT

Instrument calibration should be documented on a Field Instrument Calibration Record and in

the field logbook.

8.0 QUALITY CONTROL/QUALITY ASSURANCE

No additional procedures are required.

SOP No. S-13

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
BOREHOLE ABANDONMENT

wood.

STANDARD OPERATING PROCEDURE

BOREHOLE ABANDONMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes guidelines and procedures for field personnel to use in the supervision of borehole or soil boring abandonment and groundwater monitoring well abandonment activities. Additional specific borehole and well abandonment procedures and requirements will be provided in the project work plans.

2.0 DEFINITIONS

Borehole Abandonment – The process whereby boreholes or soil borings are grouted or sealed following completion of drilling, sampling and/or logging.

3.0 PROCEDURE

This section contains responsibilities, procedures and requirements for borehole abandonment. Abandonment procedures to be used at a particular site must incorporate project-specific regulatory requirements. Consequently, the project work plans will identify the following:

- Abandonment objectives,
- Boreholes to be abandoned,
- Specific procedures for borehole abandonment beyond those covered in this SOP; and,
- Applicable site-specific regulatory requirements for borehole abandonment.

3.1 Responsibilities

Project Manager

The Project Manager is responsible for ensuring that all abandonment activities are conducted and documented in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

Field Operations Lead

The Field Operations Lead is responsible for periodically observing field activities and review of field generated documentation associated with this SOP. The Field Operations Lead is also responsible for the implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to the abandonment requirements, issuing non-conformances, etc.) if problems occur.

Field Personnel

Field personnel assigned to borehole and well abandonment activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate

procedures. All staff are responsible for reporting deviations from the procedures to the Project Manager or Field Operations Lead.

3.2 Abandonment of Boreholes

After drilling, logging and/or sampling, boreholes should be backfilled by the method required by the applicable agency and described in the project work plans. This typically consists of backfilling to the surface with bentonite chips, pellets or bentonite-cement grout. If bentonite chips or pellets are used, they should be added to the borehole in two foot lifts and hydrated with water from a potable water supply. This process should be repeated until the entire borehole is plugged using no less than five gallons water per ten feet of borehole. If bentonite grout is used the following guidelines should be followed:

- Bentonite should be thoroughly mixed into the grout and within the percentage range specified in the work plans. If not otherwise specified in the work plans, the cement-bentonite grout mixture should be of the following proportions: 94 pounds of Portland cement, 5 pounds of powdered bentonite and a maximum of 8 gallons of water. The grout is usually tremied into the hole; however, for selected boreholes (e.g., shallow borings well above the water table) at certain sites, the grout may be allowed to free fall. In either case, care must be taken to ensure the grout does not bridge, forming gaps or voids in the grout column.
- The volume of the borehole should be calculated and compared to the grout volume used during grouting to aid in verifying that bridging did not occur.
- When using a tremie to place grout in the borehole, the bottom of the tremie should be submerged into the grout column and withdrawn slowly as the hole fills with grout. If allowing the grout to free fall (and not using a tremie), the grout should be poured slowly into the boring. The rise of the grout column should also be visually monitored or sounded with a weighted tape.
- If the method used to drill the boring utilized a drive casing, the casing should be slowly extracted during grouting such that the bottom of the casing does not come above the top of the grout column.
- During the grouting process, the drilling hands performing the task should be supervised to assure that potentially contaminating material (oil, grease, or fuels from gloves, pumps, hoses, et. al) does not enter the grout mix and that personnel are properly wearing personal protective equipment as specified in the Site Safety and Health Plan (SSHP).
- Following grouting, barriers should be placed over grouted boreholes as the grout is likely to settle in time, creating a physical hazard. Grouted boreholes will typically require at least a second visit to "top off" the hole.
- The surface hole condition should match the pre-drilling condition (asphalt, concrete, or smoothed flush with native surface), unless otherwise specified in the project work plans.

4.0 REFERENCES

MEDEP, 2009. Maine Department of Environmental Protection, Bureau of Remediation and Waste Management, Division of Technical Services, "Guidance for Well Boring Abandonment", January 7, 2009.

Maine Department of Environmental Protection

Bureau of Remediation and Waste Management

Division of Technical Services

Guidance for Well and Boring Abandonment

January 7, 2009

Below is guidance for the abandonment of wells and borings associated with environmental or geotechnical investigations.

1.0 APPLICABILITY
These guidelines are applicable for the abandonment of geotechnical borings, environmental monitoring wells and soil borings installed in the State of Maine. These guidelines pertain to any vertical or high angle boring completed by rotary or direct push methods.

2.0 PURPOSE
The purpose of these procedures are to: 1) prevent the possibility of abandoned wells and/or borings providing a means for contaminants to enter the groundwater; 2) prevent the possibility of personal injury; and 3) prevent the intermixing of separate water bearing zones.

Below are the types of wells/borings these procedures pertain to:

a) soil and rock borings that need to be properly abandoned so that they do not serve as a preferential conduit for contaminants;

b) monitoring wells that need to be abandoned after the site rehabilitation activities are completed or after the well is determined to be no longer useful so that the well can not be used in the future for unauthorized access; and

c) drinking water wells that need to be abandoned if they become unusable due to the presence of contaminants.

3.0 RESPONSIBILITES
The onsite inspector is responsible for verifying that well/borehole abandonment has been conducted in a manner that is consistent with the methods described below and meets the purpose of these guidelines as state in Section 2.0 above.

Specifically, the onsite inspector must:

- verify and document the depth and diameter of the boring/well to be abandoned;
- review available boring logs and well construction diagrams and determine and document the appropriate abandonment method;
- verify and document the type and volume of sealing material that is to used by the well/boring abandonment subcontractor;
- verify and document that the bentonite/grout slurry has been mixed to manufactures specifications;
- document the abandonment procedures, including volume of material used, incidences of bridging, corrective actions taken and end result;

- provide a signed copy of the well abandonment record to the appropriate DEP project manager.

4.0 GUIDELINES AND PROCEDURES

Whenever there is doubt as to whether the well/boring will serve as a preferential pathway for the migration of contaminants, the boring must be hydraulically sealed.

The method used for abandonment of the well/boring must be indicated in field notes or on the boring log. A well abandonment record is included with this guidance and it is recommended that the form be completed at the time of abandonment and submitted to the site-specific project manager at the Maine Department of Environmental Protection for inclusion into the permanent site file.

Abandoned wells or boreholes should be sealed in a manner appropriate to prevent the entry of contaminants and from the mixing of waters from separate water bearing zones. Also, the abandoned well/boring should not be a physical hazard to any person walking, driving or operating equipment nearby. Below are procedures specific to each well/boring scenario.

Soil borings: Abandoning soil borings with a hydraulic seal is not always necessary. For sites that have a very shallow water table (i.e.,<10'), or where soils are contaminated down to the water table, or where the lithology consists of homogeneous sand, less rigorous abandonment methods may be used. In these instances, it is acceptable to backfill the boring with soil that has permeability equal to or lower than the soil in which the boring was completed.

Conversely, a soil boring needs to be abandoned with a hydraulic seal if there is a chance, by leaving it unsealed, that mixing of waters of separate water bearing zones and/or creation of a contaminant migration pathway will result. Specifically, the soil boring needs to be sealed if:

1. the soil boring is suspected to have penetrated a perched zone or a confining or semi-confining layer;
2. more than one geologic unit is encountered (e.g., glacial marine/esker);
3. the site has a deep water table and the lithology consists of heterogeneous sands and clay;
4. soil contamination is identified, but the groundwater is not suspected of being impacted by the contamination;
5. the soil boring goes into the water table and has the potential to short circuit the hydraulic head from deeper zones; and
6. the soil boring is drilled blind (i.e., no samples collected).

Soil borings can be completed via direct push, hollow stem auger, solid stem auger and drive and wash methods. Direct push and solid stem augers require

similar abandonment methods, as do hollow stem auger and drive and wash borings.

Type A borings - For Geoprobe® type drilling that uses only the Macro-Core® to advance the hole, or when solid stem augers are used to complete the borehole, the boring will be backfilled with hydraulic sealing material after the drilling tools are removed. Sealing is completed by slowly pouring bentonite chips/pellets or other plugging material in the borehole from the top. The seal should be brought up to one foot below grade. The last foot of the boring should be backfilled with soil to avoid the mess associated with bentonite on the ground surface. It is not acceptable to use alternating layers of sand and bentonite to seal the borehole. Tremie method of grouting is acceptable, although not required for soil boring completed with non-dual wall direct push or solid stem methods.

Type B borings - For borings completed using hollow stem auger, drive and wash, or Geoprobe® type drilling that uses a dual wall sampling system, the borehole should be hydraulically sealed while the drilling tools remain in the boring.

Method 1 - The soil boring should be tremie grouted beginning from the boring bottom and continuing upward to the ground surface. The plugging material should consist of either:

1. cement with a 2% - 5% by weight bentonite mixture; or
2. high solids bentonite grout.

The cement/grout mixture should be mixed according to manufacturer's specification to produce a flowable (i.e., pumpable) consistency. The minimum volume of grout should be equal to that of the well bore. The well bore should be filled with grout to within 2 feet of the top of the cut down casing. The last 2 feet of casing should be filled with cement or clean soil to ensure a solid surface exists at ground surface.

Method 2 – The soil boring can be abandoned by pouring bentonite or other plugging material in from the top of the hollow stem augers, or casing, when drive and wash or dual wall Geoprobe® type equipment is used. This method requires care and patience because it is susceptible to bridging within the auger and casing. Bentonite chips/pellets should be added to the borehole slowly. As each lift of bentonite is added, the auger or casing must be pulled up a corresponding height to prevent bridging within the auger/casing. Continuous measuring of the seal height within the borehole is necessary as each lift of bentonite is added to ensure bridging doesn't occur. If bridging does occur, the driller must clear the bridge without removing the drilling tools from the borehole. The sealing material should be added to the borehole until it is within one foot of the ground surface. The last one foot of borehole should be filled with clean soil to avoid the mess

associated with bentonite on the ground surface. It is not acceptable to use alternating layers of sand and bentonite to seal the borehole.

Rock corings:
The rock core should be tremie grouted beginning from the well bottom and continuing upward to the ground surface. Due to the small diameter typical of rock cores, pouring bentonite chips, or other plugging material from the top down is not recommended. The plugging material should consist of either:

1. cement with a 2% - 5% by weight bentonite mixture; or
2. high solids bentonite grout.

The cement/grout mixture should be mixed according to manufacturer's specification to produce a flowable (i.e., pumpable) consistency. The minimum volume of grout should be equal to that of the well bore. The well bore should be filled with grout to within 2 feet of the top of the cut down casing. The last 2 feet of casing should be filled with cement to ensure a solid surface exists at ground surface.

Monitoring wells: There are two basic types of monitoring wells: bedrock and overburden. Abandonment method for each depends on how the wells were installed. Proof of well construction in the form of a boring log and construction diagram is required to determine whether a well was properly installed.

Type A wells - If it can be demonstrated that a well was properly constructed, meaning that hydraulic seals were placed to prevent hydraulic short-circuiting between separate water-bearing zones (i.e., when breaching a confining layer or a continuous section of un-fractured bedrock), the well screen and riser can be abandoned in place by either of following methods:

Method 1 - The well should be tremie grouted beginning from the well screen bottom and continuing upward to the ground surface. The plugging material should consist of either:

1. cement with a 2% - 5% by weight bentonite mixture; or
2. high solids bentonite grout.

The cement/grout mixture should be mixed according to manufacturer's specification to produce a flowable (i.e., pumpable) consistency. The well should be filled with grout to within 2 feet of the top of the cut down casing. The last 2 feet of casing should be filled with cement or clean soil to ensure a solid surface exists at ground surface. The surface casing should be cut down to grade (preferably below).

Method 2 – If the well casing is at least 2 inches in diameter, the well can be abandoned by pouring bentonite or other plugging material in from the top of the

well casing. This method requires care and patience because it is susceptible to bridging. Bentonite chips/pellets should be added to the borehole slowly. Continuous measuring of the seal height within the borehole is necessary as each lift of bentonite is added to ensure bridging doesn't occur. It is acceptable as a cost-saving measure, to use alternating layers of sand and bentonite to seal the well riser. For well screens that are 10 feet in length or less, an instance of bridging must be noted by the onsite inspector. As long as the riser above the screen is sufficiently sealed from the potential of surface leakage, it is not necessary to fully seal the screened interval. However, if bridging occurs in bedrock monitoring wells with screens longer than 10 feet prior to fully sealing the screened interval, the well must be drilled out and sealed via the tremie method.

Type B wells – If it can not be demonstrated that a well was properly constructed regardless of whether it is installed in the bedrock or overburden, the well must be drilled out and sealed via the tremie method.

> *Note: Methods of drilling out may include pulling the well out, re-drilling, overdrilling, etc. as long as the sand pack or other non-sealing material is removed and the seal will directly contact native material.*

Drinking water wells: Typical drinking water wells are 6-inch diameter drilled bedrock boreholes. Pumps, drop pipes, wires and all apparatus within the well casing must be removed from the well. Well depth and diameter must be measured in order to calculate the necessary volume of plugging material. The surface casing should be cut down to grade (preferably below).

Method 1 - The preferred method of well abandonment is by tremie grouting. The well bore should be tremie grouted beginning from the well bottom and continuing upward to the ground surface.

The plugging material should consist of either:

1. cement with a 2% - 5% by weight bentonite mixture; or
2. high solids bentonite grout.

The cement/grout mixture should be mixed according to manufacturer's specification to produce a flowable (i.e., pumpable) consistency. The minimum volume of grout should be equal to that of the well bore. The well bore should be filled with grout to within 3 feet of the ground surface. The remaining casing should be cut down to below grade. The remaining portion of open casing should be topped off with Portland cement. The remaining portion of the boring should be filled with clean soil to ensure a solid surface exists at ground surface.

> *Note: If possible, it is recommended that the casing be removed from the borehole following tremie grouting.*

Method 2 – A less preferred alternative method of well abandonment involves pouring bentonite chips, or other plugging material into the well from the top down. This method should be used with extreme care and patience to avoid bridging of the bentonite. If this method is to be used, it will need to be demonstrated (via field notes) that the entire borehole is filled with bentonite. It is not acceptable to use alternating layers of sand and bentonite to seal the borehole. If the volume of bentonite chips installed is less than the volume of the well bore, the well bore must be re-drilled to clear the bridge. Well abandonment will be considered by the Department to be successfully complete only if is can be demonstrated that there is a continuous seal throughout the borehole (via volume calculation comparison). Bentonite chips/pellets should be added to the borehole 1-Cup at a time. Continuous measuring of the seal height within the borehole will be required as each bag of bentonite is added to the borehole to ensure bridging doesn't occur. Voids spaces caused by bridging are not acceptable.

(Note: To avoid bridging in a 6-inch diameter well, it may be beneficial to use a 2" drop pipe, such as PVC threaded monitoring well riser. The 2" threaded riser sections are lowered to just above the bottom of the well. Bentonite chips/pellets are added slowly to the drop pipe. The pipe is slowly removed from the borehole as more bentonite is added. If a bridge does occur, it will likely occur within the riser. Once bridged, the riser is removed from the borehole and the section containing the bridge is removed and replaced with a new section of pipe. The 2" pipe is lowered back into the borehole and the process of adding bentonite resumes until the borehole is completely filled.

WELL ABANDONMENT RECORD
Maine Department of Environmental Protection
Bureau of Remediation and Waste Management

1. **Date of Abandonment**:_____

2. **Abandonment Contractor**:

_____
Company

_____
Printed Name of Individual Abandoning Well

3. **Well Location**:

_____
Address

_____
County

_____
Nearest Town

_____
Latitude (D,M,S or DD format)

_____
Longitude

4. **Well Type**:

□ direct push □ drilled

5. **Well Use**:

□ monitoring □ residential □ industrial

□ injection □ recovery □ geotechnical

6. **Reason for abandonment**:

_____

_____

_____

_____

7. **Are boring logs available?**

□ Yes, attached □ No

8. **Are well construction logs available?**

□ Yes, attached □ No

9. **Well Depth (ft)**:_____

10. **Boring Diameter (in)**:_____

11. **Riser/Casing Diameter (in)**:_____

12. **Type of Casing**:

□ steel □ PVC

13. **Was any casing removed?**

□ Yes □ No

If yes, length removed (ft.):_____

14. **Was well abandoned in place?**

□ Yes □ No

If yes, was casing perforated?

□ Yes □ No

15. **Abandonment Material**:

□ bentonite grout □ dry bentonite

□ cement grout □ native soil

16. **Quantity of Material Used**:

# of bags_____, or

cubic feet _____

17. **Explain Method of Material Placement**:

_____

_____

_____

_____

_____

18. **Signature of Person Abandoning the Well**:

_____

SOP No. S-14

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
SURFACE WATER SAMPLING

STANDARD OPERATING PROCEDURE

SURFACE SOIL SAMPLING

1.0 SCOPE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the methods used for obtaining surface water samples for laboratory chemical analysis and associated water quality field measurements. This procedure applies to all Wood Environment & Infrastructure Solutions (Wood) personnel and subcontractors with the responsibility for determining water quality and for the collection, preparation, preservation, and submittal of surface water samples for laboratory analyses.

Collection of surface water samples for laboratory analysis of organic and inorganic parameters may require specially prepared and preserved containers. This SOP also describes the procedures for using the various types of sampling equipment, which include dipping using sample container, scoops, peristaltic pumps, discrete depth samplers, bailers, buckets, and submersible pumps. The equipment may be constructed of special materials (e.g., stainless steel, Teflon® (PTFE), high density polyethylene (HDPE), borosilicate glass) according to site specific project requirements.

Select appropriate sample containers depending upon the analyses to be performed as described in the project Sampling and Analysis Plan (SAP) and Quality Assurance Project Plan (QAPP).

2.0 RESPONSIBILITIES

The field sampling personnel will be responsible for the proper use and maintenance of all types of equipment used for obtaining surface water samples, and the collection, labeling, handling and storage of all samples until further chain of custody procedures are undertaken.

3.0 HEALTH AND SAFETY WARNINGS

Proper safety precautions must be observed when collecting surface water samples. Collection of surface water samples may require working on, in, or near a water body, appropriate personal protective equipment (PPE) may include the use of approved personal flotation devices (PFD) and the buddy system. Refer to the Site Safety and Health Plan (SSHP). Additionally, sample preservatives commonly include strong acids and bases. Care should be taken to prevent human contact with the acids and bases, and in addition, particular care should be taken to store each sample separate from the other.

4.0 EQUIPMENT AND SUPPLIES

4.1 Dipping Using Sample Container

When the surface water source is directly accessible by wading or through other means, samples

may be collected by directly dipping the sample container into the surface water source. Sample personnel should face upstream if there is a current present and collect the sample without disturbing bottom sediment. Care must be taken to not displace the sample preservative from a pre-preserved sample container, such as a 40 milliliter (ml) volatile organic compound (VOC) vial containing hydrochloric acid preservative.

4.2 Scoops, Long Handled Dippers, Swing Samplers

Constructed of inert materials (stainless steel, HDPE, or PTFE), scoops, dippers and swing samplers allow the sampler to collect a surface water sample from the shoreline or other sampling platform (i.e.; boat or dock) when wading is not feasible.

Scoops should be cleaned prior to use, by following procedures outlined in SOP S-3 "Decontamination of Field Equipment". Prior to the collection of a surface water sample, the scoop should be rinsed three times with surface water from the sample location. Care should be taken to not allow the rinse water to be returned to the sample location. Once collected, the surface water sample is slowly poured directly into the sample containers. If one scoop volume is insufficient to fill the sample containers, multiple scoops of sample can be collected and used to fill the appropriate sample containers.

Long handled dippers (LHD) comprise handles of up to twelve feet in length which are attached to a plastic beaker. Beaker volumes can vary from 100 ml to 500 ml and should be constructed of an inert material. LHD samplers are used in a similar manner to scoops, however, the extended handle length allows field personnel access to sample locations without the use of an additional sampling platform such as a boat.

Swing samplers are a modified version of the IHD. The swing sampler has a jointed platform attached to the end of the long handle. The platforms allows the field sampler to secure a sample container to it using a compression ring or zip tie. The sample container is then dipped into the surface water for collection. The jointed platform allows the sample container to be placed into difficult to reach areas. Preservation of the sample should be done immediately post collection as swing samplers are typically used to collect surface water samples from fast flowing locations.

4.3 Peristaltic Pumps

A peristaltic pump can be used to sample a discrete water column interval, from a shallow pond or stream. Sample tubing should consist of inert materials such as HDPE or PTFE. The pump head tubing is flexible, such as silastic, because of the pump mechanism. An inert piece of pipe or conduit (stainless steel) is attached to the end of the sample tubing and lowered to the sample interval depth. The peristaltic pump is turned on and the flow rate is adjusted to 200 ml per minute. The pump should be run until at least three volumes of water have passed through the tube to rinse it. The discharge end of the tubing can be connected to a flow through cell and can be measured using a water quality meter (i.e., Horiba or YSI) if required.

After parameters are recorded, the discharge end of the tubing is disconnected from the flow through cell and sample containers can be filled. VOC samples should not be collected through the discharge end of the peristaltic pump tubing. If VOC samples are to be collected, they

should be collected last. The pump is shut off and the tubing is withdrawn from the sample interval. Once removed from the water column, the VOC sample container is placed under the intake end of the tubing and the water sample is allowed to drain out of the tubing either by releasing the tubing compression at the pump head, or by reversing the pump direction and adjusting the flow rate slowly discharge the water sample (50 to 100 ml/minute). This process can be repeated multiple times until all VOC sample containers are filled.

4.4 Discrete Depth Samplers

A stainless steel bomb sampler can be used to collect surface water samples in lakes and ponds. Bomb samplers typically range in volume from 4 ounces (oz.) to 32 oz. The bomb sampler remains closed and is lowered to the sampling depth using a retrieval line attached to the upper end of the bomb via an eye ring. The bomb is filled by either bouncing the end on the lake or pond bottom (if sampling over a hard bottom location) or pulling on a trigger line attached prior to deployment. Bouncing the bomb on the bottom of the lake or pond is not typically recommended as this procedure stirs up sediment which is then captured during the filling process. The trigger line prevents this from occurring and can be used to collect surface water samples from mid-depths. Bombs should be cleaned prior to sampling and between sample locations, by following procedures outlined in SOP S-3 "Decontamination of Field Equipment".

4.5 Bailers

PTFE or stainless steel bailers may also be used for surface water sample collection. The bailer is lowered into the water column using a retrieval line that is attached to the top. As the bailer is lowered into the water column, water is continually displaced through the bailer until the sample interval is reached. When the bailer is retrieved, the check valve closes at the bottom and the water interval is captured. The surface water sample is transferred to sample containers by slowly pouring the bailer contents out of the top of the bailer. Bailers can be dedicated to the sample location or re-used between sample locations. Bailers should be cleaned prior to sampling and between sample locations, by following procedures outlined in SOP S-4 "Decontamination of Field Equipment".

4.6 Buckets

A stainless steel bucket can be used to collect surface water samples when the sample location is not accessible to wading or working from another sample platform such as a boat or dock. The surface water sample location is typically accessed from a bridge. A retrieval line is tied to the handle of the bucket which is then lowered to the water's surface and allowed to fill. Once retrieved, samples can be poured out of the bucket into the appropriate sample containers. The bucket method can also be used to record water parameters using a water quality meter.

Extra precautions should be used while working from a bridge. Bridge work typically involves working at an elevated location and may be associated with vehicular traffic.

4.7 Submersible Pumps

Submersible pump components that come into contact with the surface water sample should be constructed of inert materials such as stainless steel and PTFE. Samples can be collected directly into the appropriate sample containers. Submersible pumps are re-useable and should be

cleaned prior to use by following procedures outlined in SOP S-3 "Decontamination of Field Equipment".

Submersible pumps can be used to collect discrete samples by placing the pump at the sample depth desired, or to collect composite samples. Composite samples are collected by raising and lowering the submersible pump throughout the water column. Composite samples should be pumped into an inert, large volume container, such as a stainless steel bucket. Once the sampling activity is completed, the composited sample can be poured off into the appropriate sample containers.

5.0 DOCUMENTATION

Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms include:

1. Field Logbooks
2. Surface Water and Sediment Sampling Record

Refer to the SAP and QAPP for required documentation procedures.

6.0 REFERENCES

USEPA, 2013. Region 4, U.S. Environmental Protection Agency, Science and Ecosystem Support Division, Operating Procedure "Surface Water Sampling", SESDPROC-201-R3, Revision 3, February 28, 2013.

SOP No. S-15

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
ELEMENTAL ANALYSIS USING THE INNOV-X SYSTEMS
FIELD X-RAY FLUORESCENCE ANALYZER (XRF)

wood.

STANDARD OPERATING PROCEDURE

ELEMENTAL ANALYSIS USING THE INNOV-X SYSTEMS

FIELD X-RAY FLUORESCENCE ANALYZER (XRF)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedures used to analyze soil samples for metals using the INNOV-X Systems Alpha 4000 portable X-ray Fluorescence (XRF) analyzer. This SOP should be used in conjunction with the INNOV-X Systems XRF Manual.

EPA method 6200 will be used to analyze soil and sediment samples using the XRF. A listing of elements and reporting limits of metals analyzed by the XRF is presented in table 1 of this SOP. The following documents were used to prepare this SOP for elemental analysis at Sites:

- USEPA Method 6200. Field Portable X-ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment. January 1998.
- Region I, EAP-New England. Standard Operating Procedure For Elemental Analysis Using the X-MET 920 Field X-ray Fluorescence Analyzer. USEPA Region I Quality Assurance Unit Staff. October 1996.
- Innov-X Systems, Inc. Metals in Soil Analysis Using Field Portable X-Ray Fluorescence. January 2003.
- Innov-X Systems, Inc. Alpha 4000 Analyzer User Manual. Innov-X Systems, Inc. Version 1.1. October 2002.

2.0 METHOD SUMMARY

2.1 Principles of Operation

XRF is a nondestructive qualitative and quantitative analytical technique used to determine the chemical composition of metals in a sample. In an XRF analysis, primary X-rays emitted from an X-ray tube or a sealed radioisotope source are utilized to irradiate a sample. The primary X-rays incident on the sample cause the elements present in the sample to emit (that is, fluoresce) their characteristic X-ray line spectra. The elements may be identified by the energies of the wavelengths of their spectral lines. The unit of energy of an X-ray is the kiloelectron volt (keV). The X-ray energy is proportional to the frequency of the X-ray waves and is inversely proportional to the wavelength. Since it is a fluorescent process, the energy of the fluorescent X-rays will always be of lower energy than the primary X-ray energy. In addition to the fluorescent X-rays, there will be a backscattering of the primary X-rays. Energies of the fluorescent and scattered X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are

linearly proportional to the energy. An electronic multichannel analyzer measures the pulse amplitudes which is the basis of qualitative X-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis.

2.2 Sample Preparation and Analysis Summary

For quantitative analysis of soil; sticks, stones, and other matter that is non-representative of the sample are removed, and the sample is thoroughly homogenized. There are three accepted methods of sample analysis:

1. In-situ direct: a sample reading is taken directly from the sample location
2. Ex-situ unprepared: a sample is collected into a plastic bag, non-representative material is removed (sticks, stones, non-soil material, etc.) and a reading is taken using the bag as a sample container
3. Ex-situ prepared: a sample is collected into a plastic bag or glass soil jar, non-representative material is removed (sticks, stones, non-soil material, etc.), an aliquot of the collected sample is then prepared as described below, and a reading is done on the prepared sample.

The sample is dried in an oven at 150^{o}C for 2 to 4 hours and then sieved through a No. 60 mesh sieve. If the sample exhibits a high clay content (clumping), it is ground up using a mortar and pestle prior to sieving. The fraction of the sample that passes the No. 60 sieve is then re-homogenized and transferred into the XRF sample cup.

The sample cup is capped with a clear Mylar film and the sample identification is clearly labeled prior to analysis. Refer to section 11.0 for a complete discussion of sample preparation.

For analysis, the cup is positioned on top of the XRF analyzer and exposed to primary X-rays from the selected radiation source. The sample fluorescent and backscatter X-rays are detected and the results are recorded by the data system. Qualitative determinations of the elements present in the sample are based on the locations of characteristic peaks produced by individual elements in the energy spectra. Quantitative determination of an element present is made by comparing the intensity of a characteristic peak in the sample to a calibration curve of the same peak developed from standards of similar matrix and known concentrations.

3.0 DEFINITIONS

- SRM - Standard Reference Material
- FPXRF - Field Portable XRF instrument

4.0 HEALTH AND SAFETY

The INNOV-X XRF analyzer uses an X-ray tube to generate ionizing radiation for sample analysis. During all measurements the sample cup must be positioned on the analyzer

so that the sample cup shields the analyst from exposure to radiation. The probe must not be opened except by authorized personnel. Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to field operations. Radiation safety information for the INNOV-X XRF can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local, state and national regulations that pertain to the use of radiation-producing equipment. Radiation safety guidelines for the instrument used the Site are presented in section 3.2 of the INNOV-X Systems Instrument User Manual – Recommended Radiation Safety Training Components. A radiation monitoring program using TLD film badges will be used at the Site. All reasonable measures, including labeling, operator training, and the concepts of time, distance, and shielding, will be implemented to limit radiation exposure to *as low as reasonably achievable* (ALARA).

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Chemical Matrix Interferences

An interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another. If the XRF is calibrated for both elements (CASE 1) i.e. the one causing the interference and the one being interfered with, it is generally capable of correctly handling the interference. In this case, the element being interfered with may be measured with a poorer detection limit or poorer precision, but the analytical results should still be acceptable for field-portable XRF. If the XRF is not calibrated for the element causing the interference (CASE 2), then the XRF may report the presence of elements not in the sample, or greatly elevated concentrations of elements in or not in the sample.

- Example CASE 1: Lead and arsenic. Most XRFs are calibrated for lead and arsenic. Lead interferes with arsenic (not vice-versa though). The net effect is a higher detection limit for arsenic, and poorer precision. The XRF handles the correction automatically, but the precision is affected. The loss of precision is also reported by the XRF. (Please refer to Innov-X Applications Sheet: *In-field Analysis of Lead and Arsenic in Soil Using Portable XRF* for more detail).
- Example CASE 2: Bromine in the sample, but XRF is not calibrated for bromine. Bromine, as a fire retardant, is being seen more and more in soil and other sample types. For this reason, Innov-X analyzers include Br in the calibration data. If Br is not calibrated, but is present in the sample, the analyzer will report highly elevated levels of Pb, Hg and As. The levels will depend upon the concentration of Br in the sample.

Interferences between elements can be broadly categorized into a) Z, Z-1, Z+1 interferences, and b) K/L interferences. Interference type "a" occurs when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 or Z+1. Generally, portable XRFs have good correction methods,

so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe

(Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 or Z=27 respectively). The type "b" interference occurs when the L-shell line of one element overlaps with the K-shell spectral line of another element. The most common example is the lead/arsenic interference where the L-alpha line of lead is in nearly the exact same location as the K-alpha line of arsenic.

5.2 Moisture Content

Sample moisture content will affect the accuracy of the sample results. The measurement error may be minor when the moisture content is small (5 to 20 %), or it may be significant when measuring the surface of soils that are saturated with water. For quantitative analysis, moisture content will not be an issue because all samples are dried as part of the sample preparation.

6.0 PERSONNEL QUALIFICATIONS

Sample analysis will be performed by an authorized user. The authorized user must be thoroughly familiar with the Wood radiation Protection Program, this SOP, and the Innov-X Systems XRF Reference Manual supplied by the instrument manufacturer.

7.0 EQUIPMENT AND SUPPLIES

7.1 Innov-X Systems Alpha 4000 instrument and accessories

- Alpha 4000 Analyzer with iPAQ attached.
- (2) lithium ion batteries.
- Batter charger and AC adaptor.
- Standardization cap.
- iPAQ cradle and AC adapter.

7.2 Computer

- Excel program for recording data in spreadsheet format.

7.3 Supplies

- Ziploc, quart sized plastic bags for sample collection
- 2 oz glass soil jars for offsite split samples (QC clean quality)
- Oven – for drying sediment and soil samples.
- Sieve – No. 60 (250 µm) stainless steel.
- Polyethylene XRF sample cups – purchased from SPEX Sample Prep, LLC. Cat# 3529 (x-ray cell with snap ring, 31mm).
- Mylar film for sample containment, 2.5 or 6.0µm thick.

- Stainless steel spatulas.
- Mortar and pestle (ceramic or glass).
- Aluminum drying pans.
- Gloves.
- Safety glasses.
- Portable hood.
- Run log book (to record sample analyses).
- NIST SRM – For instrument calibration checks (SRM 2709, SRM 2710 and SRM 2711).
- Instrument Blank standard provided by Innov-X Systems.
- Silicon dioxide (SiO_2) 99.995% clean – for method blank analysis.

8.0 CALIBRATION AND OPERATION

Procedures for calibration and operation of the Alpha 4000 Analyzer are taken from EPA Method 6200 and updated to be specific to the Innov-X analyzer.

The XRF instrument will be calibrated at the factory prior to delivery at the Site. The Alpha 4000 Analyzer will be calibrated by Innov-X Systems Inc. using the Compton Normalization method consisting of the analysis of a single, well characterized standard, such as an SRM or SSCS. The standard data are normalized to the Compton peak. The Compton peak is produced from incoherent backscattering of X-ray radiation from the excitation source and is present in the spectrum of every sample. The matrix affects the way in which source radiation is scattered off the samples. This scatter is directly related to the intensity of the Compton peak. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

Operation of the Alpha 4000 Analyzer at the Site will performed as described in the Innov-X Systems User Manual Version 1.1, October 2002.

9.0 QUALITY ASSURANCE AND QUALITY CONTROL

The following section details proper quality assurance is detailed for analysis of sediment and soil samples using the XRF analyzer. All operators will perform QA/QC procedures as described in this SOP. Procedures are listed below:

9.1 Proper Verification of Instrument Operation

The following procedures were taken from USEPA Method 6200 and updated to be specific to the Innov-X analyzer. Quality assurance here consists of testing known standards to verify calibration, as well as testing blank standards to determine limits of detection and to check for sample cross contamination or instrument contamination.

Components of instrument QC:

1. ENERGY CALIBRATION: An energy calibration check sample will be analyzed at the beginning of each day. The Innov-X analyzer performs this automatically; this is the purpose of the standardization check when the analyzer is started. The software does not allow the analyzer to be used if the standardization is not completed. The energy calibration check is performed by placing the snap on metal clip on the front of the analyzer and selecting standardize on the analysis screen. If the energy calibration fails, the analyst will shut down the instrument, replace the battery with a fully charged back up, and restart the instrument. An energy calibration will be performed after restarting the XRF.

2. INSTRUMENT BLANK: An instrument blank will be analyzed at the beginning of each day, and for every 20 environmental samples. The operator should use the silicon dioxide (SiO2) blank provided with the analyzer. The purpose of this test is to verify there is no contamination on the analyzer window or other component that is "seen" by the x-rays. Method 6200 recommends an instrument blank at least once per day, preferably every 20 samples. For either in-situ or prepared-sample testing, the operator should test the SiO2 blank to be sure there are no reported contaminant metals. If target analytes are reported in the instrument blank, all contact surfaces of the instrument will be wiped down with a soft cloth to remove any contamination on the detector window (the instrument blank should also be wiped down to ensure it has not been contaminated, or a different instrument blank may be used, Teflon® or quartz block). If the instrument continues to detect target analytes in the instrument blank, the Kapton® window covering the detector should be replaced.

3. METHOD BLANK: A method blank will be analyzed daily or for every 20 prepared samples. The purpose of the method blank is to verify that cross-contamination is not introduced into samples during the sample preparation process. A method blank will be prepared with each batch of 20 samples (Method 6200 recommends following the sample preparation procedures with clean SiO2 once very 20 prepared samples). If target analytes are detected in the method blank, all sample prep equipment should be thoroughly cleaned and all samples prepped under that blank should be evaluated. An action limit of five times the reported blank concentration will be established. Any sample results greater than the action limit will be accepted. Sample results below the action limit will require re-prepping and re-analyzing the affected samples after the preparation equipment have been thoroughly cleaned.

4. CALIBRATION VERIFICATION: A calibration verification check (NIST SRM check standard) will be analyzed at the beginning of each day, after 20 samples have been analyzed, or every 4 hours, whichever is more frequent. A calibration verification standard should be selected with target analyte concentrations less than, at/near, and greater than the project action limit. Each calibration verification standard should be monitored, in turn, throughout the field program, this will provide a check on instrument performance overall. The operator will perform a 2-minute test on a NIST standard. The percent difference (%D) between the FPXRF result for an element and the value of the

standard should be 20 or less. If the calibration check is greater than 20% of the standard value, the operator will adjust the calibration factor of the instrument and re-analyze the standard (see instrument manual for re-adjustment of calibration factors).

5. LABORATORY DUPLICATE: A laboratory duplicate sample will be analyzed daily. The laboratory duplicate is prepared and analyzed in duplicate with the original sample. The project control limit for the laboratory duplicate relative percent difference (RPD) between the original sample and lab duplicate sample is 50, when positive results for both samples are ≥ 5 times the quantitation limit. If the laboratory duplicate RPD exceeds the 50, sample preparation techniques (specifically homogenization prior to collection of the raw sample aliquot for drying) will be evaluated and improved, if necessary.

6. PRECISION MEASUREMENTS: The precision of the method is monitored by analyzing samples with target analyte concentrations less than, at or near, and greater than the project action limit. During the beginning phase of the program, after sufficient samples have been collected and analyzed for appropriate selections, one sample from each category will be analyzed in replicate seven times. Statistical analysis of the replicate samples, at each concentration category, will be performed. Statistical analysis includes calculation of the percent relative standard deviation (RSD), the standard deviation (SD), and the mean concentration. For the FPXRF data to be considered precise, the RSD for target analytes should be less than or equal to 20.

10.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

Soil and sediment samples will be collected in press seal plastic bags (Ziploc® or equivalent). Initial homogenization of the sample and removal of non-representative material should take place at the time of sampling. To maintain sample integrity, documentation of all sample locations, dates, times, depth, and associated field sample identification numbers will be recorded in field logbooks at the time of sample collection.

On-site sample documentation procedures are presented in the site Sampling and Analysis Plan (SAP). Samples may be stored at room temperature and have an indefinite shelf life.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Sample Preparation

Field samples are spread out in an aluminum drying pan, clumps are broken up with a stainless steel spatula, and the sample is oven-dried at < 150°C for 2 to 4 hours to remove moisture. After drying, all large organic debris and non-representative material (sticks, twigs, leaves, roots, insects, asphalt, rocks, etc.) are removed and the sample is transferred to a mortar and pestle and ground to a uniform consistency. The dried and ground sample is then sieved through a No. 60 (250 µm) mesh stainless steel sieve. At no time should the material be forced through the sieve. The sieved fraction is collected

on a white sheet of paper. Pebbles and organic matter remaining on the sieve should be discarded. The under-sieve fraction of the material constitutes the sample. Fill one XRF sample cup approximately 3/4 full with sample. Cut and tension (wrinkle-free) a piece of Mylar film over the top of the cup and seal using the plastic securing ring. Label the sample cups appropriately. The stainless steel sieve and spoons must be wiped clean with a paper towel between sample preparations.

Alternatively, field samples collected in press seal bags are analyzed by balling up the representative sample and placing the bag with sample upon a hard surface (table top or ground). The probe end of the XRF is then placed in contact with sample bag and held in place during the duration of the analysis interval (60 seconds). It is important that all sticks and rocks have been removed from the sample prior to analysis.

11.2 Sample analysis

Analysis of sample, blanks and check standards (SRMs) will be performed using the Innov-X Systems Alpha 4000 instrument and Innov-X Systems Analyzer software. Refer to section 4.0 of the instrument manual for sample analysis using the analyzer software.

11.3 Analysis Sequence

- Install battery in the XRF unit. Battery should remain charging overnight, when the instrument is not in use.
- Install the iPAQ unit on the top of the XRF. Turn on instrument. Allow instrument to warm-up for 1 hour prior to sample analysis.
- Perform the standardization procedure with the standardization clip attached to the front of the analyzer.
- Analyze the initial calibration check using the SRMs provided with the instrument. There are three SRMs (SRM 2709, SRM 2710 and SRM 2711) that will be analyzed. The percent difference (%D) of the calibration check standard must be < ± 20% to continue with analysis. If the %D is greater then 20, the instrument will need to be re-calibrated, per manufactures specifications.
- Analyze the instrument blank (provided with the instrument). There should be no detections greater than the reporting limits.
- Analyze the Method Blank.
- Analyze 20 samples.
- Analyze the Continuing calibration standard (SRM)
- Continue analysis of samples, analyzing a continuing calibration sample after every 20 samples, and a method blank with every batch of 20 samples. A laboratory duplicate sample is analyzed daily.

12.0 DOCUMENTATION AND REPORTING RESULTS

Sample raw results will be recorded in the field lab log book. The sample raw results will then be evaluated by the field technician for detections above the reporting limit (RL) established for the program (50 mg/kg-dry). Values less than the RL will be reported as "50U". Analysis results will also be recorded on an excel spreadsheet for loading into a database.

13.0 EXAMPLE CALCULATIONS

Percent Difference (%D)

$$\frac{\text{Known result} - \text{Determined result}}{\text{Known result}} \times 100$$

Relative Percent Difference (RPD)

$$\frac{/\ \text{Original result} - \text{Duplicate result}\ /}{(\text{Original result} + \text{Duplicate result})/2} \times 100$$

Standard Deviation (SD)

$$\sqrt{\frac{R1 + R2 + R3 + R4 + R5 + R6 + R7}{7}}$$

Mean

$$\frac{R1 + R2 + R3 + R4 + R5 + R6 + R7}{7}$$

Percent Relative Standard Deviation (RSD)

(SD/Mean) * 100

13.0 REFERENCES

USEPA Method 6200. Field Portable X-ray Fluorescence Spectrometry For The Determination of Elemental Concentrations in Soil and Sediment. January 1998.

Region I, EAP-New England. Standard Operating Procedure For Elemental Analysis Using the X-MET 920 Field X-ray Fluorescence Analyzer. USEPA Region I Quality Assurance Unit Staff. October 1996.

Innov-X Systems, Inc. Metals in Soil Analysis Using Field Portable X-Ray Fluorescence. January 2003.

Innov-X Systems, Inc. User Instruction Manual Alpha Series X-Ray Fluorescence Spectrometers. Innov-X Systems, Inc. Revision B, March 2003.

TABLE 1

On-site Metals Analysis using Innov-X Systems XRF

Medium/Matrix: *Solid*

Region I Matrix Code (from EPA-NE DQO Summary Form): *SO*

Analytical Parameter: *Metals*

Concentration Level: *Low*

Field Analytical or Fixed Laboratory Method/SOP: Field Method EPA 6200 (XRF)

Contaminants of Concern Table (Reference Limit and Evaluation Table)						
Analyte	**CAS Number**	**Project Action Limit (PAL) for soil (mg/kg)**	**Project Quantitation Limit Derived from Off-site lab methods 6010B (mg/kg)**	**XRF Instrument Estimated Level of Detection[3] (mg/kg)**	**Achievable off-site Laboratory Limits (mg/kg)**	
					MDLs	**QLs**
Lead*	7439-92-1			50 [1]		
Arsenic*	7440-38-2			50 [1]		
Lead*	7439-92-1			16 [2]		
Arsenic*	7440-38-2			10 [2]		

* - Contaminant of Concern

1 – Raw sample, no preparation

2 – Dried and sieved sample

SOP No. S-16

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE
PRIVATE DRINKING WATER WELL SAMPLING

wood.

STANDARD OPERATING PROCEDURE PROCEDURES
PRIVATE DRINKING WELL WATER SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes guidelines and procedures by which Wood Environment & Infrastructure Solutions (Wood) personnel should conduct groundwater sampling at private and public supply wells. Proper procedures are necessary to assure the quality and integrity of groundwater analytical results. Additional specific procedures and requirements will be provided in work plans and/or field work notifications, as applicable.

2.0 SCOPE

This procedure applies to all Wood personnel involved in the sampling of private and/or public water supply wells. Construction and operation of supply wells will vary; therefore, this SOP may not be applicable to all situations.

This procedure has been developed to serve as management-approved professional guidance for the Wood Program. As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment to accommodate unforeseen circumstances. Deviation from this procedure in planning or in the execution of planned activities must be approved by the Project Manager.

3.0 DEFINITIONS

Private Water Supply Well – A well that can serve as a private drinking water system, has fewer than 15 individual connections, or regularly serves an average of less than 25 individuals for less than 60 days out of the year.

Public Water Supply Well – A well and distribution system that has at least 15 service connections or regularly serves an average of at least 25 individuals daily at least 60 days out of the year. The term includes (1) any collection, treatment, storage, and distribution facilities under control of the supplier of water and used primarily in connection with the system; and (2) any collection (including wells) or pretreatment storage facilities not under the control of the supplier which are used primarily in connection with the system.

Potable Water – A liquid that is suitable for drinking.

Note: The definitions provided for private and public water supply wells are generally accepted industry-wide. However, the definitions should be confirmed with the local and state regulatory authorities where the work is being conducted. Site-specific definitions should be included in site Sampling and Analysis Plan (SAP).

4.0 PROCEDURE

This section contains both the responsibilities and procedures involved with sampling private and public supply wells. Proper procedures are necessary to insure the quality and integrity of the

samples. The details within this SOP should be used in conjunction with the site SAP. The installation-specific work plans will generally provide the following information:

- Sample collection objectives;
- Well locations to be sampled;
- Number and volume of samples to be collected at each well;
- Types of chemical analyses to be conducted for the samples;
- Specific quality control procedures and sampling required;
- Any additional sampling requirements or procedures beyond those covered in this SOP, as necessary; and
- At a minimum, the procedures outlined in this SOP for supply well sampling will be followed.

5.0 RESPONSIBILITIES

Project Manager

The Project Manager (PM) is responsible for ensuring that sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

The PM will select the appropriate sampling methodology and analytical program based on the objectives of the sampling. The PM is also responsible for ensuring that the site-specific sampling plan is clear in defining sampling methods.

Field Operations Lead

The Field Operations Lead (FOL) is responsible for periodic observation of field activities and review of field generated documentation associated with this SOP. The FOL is also responsible for implementation of corrective action (i.e. retraining personnel, additional review of work plans and SOPs, variances to QC sampling requirements, issuing non-conformances, etc.) if problems occur.

Field Personnel

Field personnel assigned to supply well sampling activities are responsible for completing tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from procedures to the Project Manager or Field Manager.

5.1 METHOD SUMMARY

The basic procedures for sampling private and public supply wells are similar to sampling of groundwater monitoring wells. The main difference is how or where the well water is accessed. Wells with in-place plumbing are commonly found at residences, and supply wells may or may not have sampling ports at the well head. The procedure can be summarized as follows.

- Decontaminate any equipment that will come into contact with water inside the well and/or sampled water as specified in SOP S-3 Decontamination of Field Equipment;

- Purge water until specific parameters have stabilized, toward ensuring formation water (as opposed to stagnant well water) will be sampled;
- Collect samples in laboratory-supplied containers; and
- Follow standard sample handling and custody procedures to contain and transport samples to the off-site laboratory.

5.2 FIELD PROCEDURES

Field procedures will incorporate other applicable project-specific SOPs, refer to the SAP.

5.2.1 PREPARATION

Office Procedures

- Contact the well owner with the proposed schedule for sampling, and coordinate with the well owner on timing; obtain information on the pumping rate and frequency during the last several weeks, if available.
- Review the work plan and the procedure including well construction, development, and sampling information on the wells to be tested, if available.
- Check out and ensure the proper operation of all field equipment.
- Assemble a sufficient number of field forms to complete the field assignment.
- Assemble appropriate testing equipment.

Equipment Selection and Sampling Considerations

This SOP assumes that private or public supply wells are equipped with operational mechanical systems to collect samples.

The following should be considered when choosing the location to collect a potable water sample from a private and/or public water supply well (EPA, 2013):

- Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest.
- Whenever possible, choose the tap closest to the water source, and prior to the water lines entering the residence, office, building, etc., and also prior to any holding or pressurization tanks.
- The sampling tap must be protected from exterior contamination associated with being too close to a sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collection procedure because it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. If the tap is too close to the ground for direct collection into the appropriate sample container, it is acceptable to use a smaller container to transfer sample to a larger container.
- When filling any sample container, care should be taken that splashing drops of water from the ground or sink do not enter into either the bottle or cap.

- Leaking taps that allow water to discharge from around the valve stem handle and down the outside of the faucet, or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations.
- Disconnect any hoses, filters, or aerators attached to the tap before sampling. These devices can harbor a bacterial population if they are not routinely cleaned or replaced when worn or cracked.
- Taps where the water flow is not constant should be avoided because temporary fluctuation in line pressure may cause clumps of microbial growth that are lodged in a pipe section or faucet connection to break loose. A smooth flowing water stream at moderate pressure without splashing should be used. The sample should be collected without changing the water flow.

Data Form

The Groundwater Sample Collection Log record (see SAP) shall be used to record sampling information and observations. All entries shall be made in indelible ink.

5.2.2 PERFORMING THE SAMPLING

Private and public supply well samples will be collected by filling sample containers from sample ports at each designated location. Ideally, the sample should be collected from a tap or spigot located at or near the well head or pump house and before the water supply is introduced into any storage tanks or treatment units. If the sample must be collected at a point in the water line beyond a tank, a sufficient volume of water should be purged to provide a complete exchange of fresh water into the tank and the tap or spigot. If the sample is collected from a tap or spigot located just before a storage tank, spigots located downstream of the tank should be turned on to prevent any backflow from the tank to the tap or spigot. Several spigots may be opened to provide for a rapid exchange of water.

The following general procedures will be used. These procedures may be modified to reflect site-specific conditions.

1. Don personal protective equipment (PPE) appropriate for the task, in accordance with the Site Safety and Health Plan (SSHP), as applicable.
2. Sample wells from least contaminated to most contaminated, if possible.

The sample port for a private well supply will be opened and allowed to flush for at least 15 minutes, when possible. The sample port for a public supply well will be allowed to flush for at least one minute. During flushing activities, collect one set of physical parameters (i.e. pH, specific conductance, dissolved oxygen, oxidation-reduction potential, turbidity and temperature). Use professional judgment for additional purging, if physical parameters do not stabilize.

3. The samples will then be collected directly from the sample port into the laboratory-supplied container. Samples should be collected with as little agitation or disturbance as possible.

Note: According to EPA (2013), "[a] well with an intermittently run pump should, in all respects, be treated like a well without a pump. In these cases, parameters are measured and the well is sampled

from the pump discharge after parameter conditions have been met. Generally, under these conditions, 15 to 30 minutes will be adequate."

6.0 QUALITY CONTROL QUALITY ASSURANCE

Quality control for this groundwater sampling method involves the collection of field quality control samples including field duplicated, matrix spike/matrix spike duplicate samples, trip blank samples, equipment blank samples, and temperature blank samples. Frequency of collection or QC samples will be specified in the SAP.

7.0 REFERENCES

U.S. Environmental Protection Agency (EPA), 2013. Science and Ecosystem Support Division (SESD) Operating Procedure: Potable Water Supply Sampling. SESDPROC-305-R3. Effective date May 30, 2013.

SOP No. S-17

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

DRILLING AND HEAVY EQUIPMENT DECONTAMINATION

wood.

STANDARD OPERATING PROCEDURE
DRILLING AND HEAVY EQUIPMENT
DECONTAMINATION

1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the methods to be used for the decontamination of drill rigs and heavy equipment which becomes potentially contaminated during a sample collection task. The equipment may include drill rigs, backhoes, augers, drill pipe, bits, casing, and screens, or any other type of heavy equipment used during field activities.

Decontamination is performed as a quality assurance measure and a safety precaution. It prevents cross- contamination between samples and also helps to maintain a clean working environment for the safety of all field personnel.

Decontamination is primarily achieved by agitation with stiff bristle brushes, rinsing with a high-pressure pump and steam-spray unit, and containment and collection of rinse materials. Equipment will be allowed to air dry after being cleaned or may be wiped dry with clean cloth or paper towels if immediate re-use is needed.

2.0 RESPONSIBILITIES

It is the primary responsibility of the project Field Operations Lead and field samplers to assure that the proper decontamination procedures are followed and that all waste materials produced by decontamination are properly stored and disposed of.

It is the responsibility of the project safety officer to draft and enforce safety measures which provide the best protection for all persons involved directly with sampling and/or decontamination.

It is the responsibility of any subcontractors (i.e., drilling contractors) to follow the proper designated decontamination procedures that are stated in their contracts and outlined in the Site Safety and Health Plan (SSHP).

It is the responsibility of all personnel involved with sample collection or decontamination to maintain a clean working environment and to ensure that any contaminants are not negligently introduced to the environment.

3.0 EQUIPMENT AND MATERIALS

3.1 Cleaning Equipment

- A remotely operated, high pressure pump with steam-spray unit is required.
- Stiff bristled brushes with various length handles and head widths.
- Plastic tubs for deconning smaller pieces of equipment such as screens.
- Polyethylene sheeting, minimum 6 mil thickness.

- Various lengths of 4x4 lumber (to construct containment berms).
- Wash bottles and manual pump sprayers.
- Assorted personal protective equipment (PPE) such as gloves, safety glasses or goggles, Tyvek® suits. Refer to the SSHP.
- Portable trash water pump and hoses.
- Portable generator.
- 55 gallon, open top drums.

3.2 Cleaning Liquids

Cleaning liquids may include tap (potable) water, deionized water, and soap and/or detergent solutions, and 10% methanol solution. For the site, only deionized water and Liquinox® detergent will be used unless otherwise specified in the site Sampling and Analysis (SAP) for a specific sampling location.

4.0 PROCEDURES FOR DECONTAMINATION

The field operations lead (FOL) will designate the equipment decontamination area. Site traffic patterns and work areas will be considered when determining where the equipment decontamination area should be established.

The heavy equipment contractor will construct a decontamination pad at the designated area, using the polyethylene sheeting and 4x4 lumber. The decontamination pad should be of sufficient area for the heavy equipment being cleaned and sufficient volume to capture the anticipated volume of cleaning fluids used. Decontamination of heavy equipment will be performed within the decontamination pad.

Heavy equipment will be transferred to the decontamination pad and cleaned between each location and prior to the initiation of use for sample collection. Steam cleaning/pressure washing water will be transferred to 55 gallon drums using the trash pump between cleanings. This will be done to prevent cross contamination of heavy equipment between sample locations.

Using the high pressure steam spray unit, spray areas (rear of rig or backhoe) that have been exposed to contaminated soils. Spray down all surfaces, including the undercarriage.

Augers, drill pipe, bits, casing, and screens will be rinsed down using the high pressure steam cleaning unit. The items should then be scrubbed using a stiff brush and soap or detergent solution. This is followed by a tap water rinse, a 10% methanol rinse (if required, refer to SAP), and finally by a deionized water rinse.

Using the trash water pump, transfer all captured liquids in the decontamination pad to a 55 gallon drum prior to removal of the heavy equipment form the decontamination pad.

Document that decontamination was completed in the appropriate field logbook.

5.0 REFERENCES

USEPA, 2013. U.S. Environmental Protection Agency, Science and Ecosystem Support Division, Operating Procedure "Field Equipment Cleaning and Decontamination", SESDPROC-205-R3, Revision 3, December 18, 2015

NIOSH/OSHA/USCG/EPA, 1985, "Occupational Health and Safety Guidance Manual for Hazardous Waste Site Activities", U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, October, 1985

SOP No. S-18

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

METHANOL EXTRACTED ROCK CHIP SAMPLING

wood.

STANDARD OPERATING PROCEDURE
METHANOL EXTRACTED ROCK CHIP
SAMPLING

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes a sample preparation procedure for the collection of fractured bedrock core samples for analysis of VOCs. Rock core samples will be extracted in the field in methanol, and an aliquot of methanol will be submitted to the laboratory for analysis. The SOP is applicable to environmental bedrock groundwater investigations.

2.0 SUMMARY OF METHOD

The analysis of methanol extracted rock chip (MERC) samples collected from fracture zones within rock core samples provides data that may be used to evaluate the potential presence of product in the fracture zones. This method of sampling is used in conjunction with rock core drilling techniques.

Rock core samples retrieved from a borehole are examined for the presence of natural hydraulically active fractures. The face of a selected fracture is chipped away using a rock hammer, chisel, or rock saw, depending on the nature of the cored bedrock fracture material. The chipped fracture face materials are containerized in a 12 ounce (oz.) sample jar containing approximately 100 ml of methanol. The sample is allowed to bathe in the methanol for approximately 12 hours. An aliquot of the methanol is then extracted from the sample jar and submitted for laboratory analysis.

3.0 DEFINITIONS

Hydraulically Active Bedrock Fracture – A fracture within the bedrock through which groundwater may flow. In bedrock core samples an active fracture will appear as a break in the rock core with some evidence of weathering, alteration, or deposition along the face of the fracture.

4.0 HEALTH AND SAFETY WARNINGS

This SOP does not present a hazard analysis specifically associated with this procedure and cannot address all hazards associated with drilling activities. Health and safety details such as activity hazard analysis should be referenced in a Site Safety and Health Plan (SSHP). The following are general health and safety guidelines associated with any drilling activity conducted at an environmental investigation site.

Rock core drilling involves the use of diesel engines, powerful hydraulics, and many moving parts and overhead objects. Potential hazards include but are not limited to pinching, crushing, piercing, repetitive motion, and noise hazards. Appropriate personal protective equipment (PPE) should be used to protect against these and other general site hazards.

Rock chip sampling involves the use of and potential exposure to methanol, which is a very volatile and flammable liquid. The use of rock chip sampling techniques are typically part of an environmental investigation. The potential exists for exposure to variable levels of site specific contaminants that may not be fully characterized.

5.0 CAUTIONS

A source of clean drilling water must be located and analyzed, so that contaminants are not introduced into the bedrock fracture system. Bedrock core drilling may require the use of large quantities of water.

It may be necessary to recirculate drilling water. If water is recirculated it must be changed frequently, particularly in fracture zones where contamination is noted or suspected.

6.0 INTERFERENCES

The methanol sample jars should not be prepared so far in advance of use that they are stored for more than 48 hours. The prepared sample jars should be stored at 4 degrees celsius and should be disposed of if exposed to any volatile vapors or fumes (i.e. gasoline, diesel).

7.0 PERSONNEL QUALIFICATIONS

Personnel using this SOP should be familiar with the methods of drilling which coincide with rock core sample collection.

Personnel using this SOP should be familiar with rock core logging techniques and be briefed on how to identify hydraulically active fractures in a rock core sample.

Personnel using this SOP should be trained to the appropriate degree in hazardous waste site safety procedures as required by OSHA regulations.

Drill operators should be qualified to operate the drill and be licensed (if applicable) in the State in which the work is being performed. Drill operators and assistants should also be trained in hazardous waste site safety procedures as required by OSHA regulations.

8.0 EQUIPMENT AND SUPPLIES

The following list is not exhaustive, but provides a listing of the minimum required equipment and supplies:

- Drill equipment – rig, core barrel, etc.
- Clean tested water supply
- pre-weighed 8 or 12 ounce clear wide mouth jars (or appropriate size to accommodate core)
- 2 mL amber vials/sample labels/tape
- Purge and trap grade methanol

- syringes and pipettes
- Balance
- Notebook/field book/rock core logs
- 6 foot folding rule
- rock hammer and cold chisel
- core boxes
- indelible markers
- stainless steel bowl
- Cooler, ice, zip lock bags, paper towels
- Chain of custody forms/seals

9.0 PROCEDURES

1. Prior to sampling, the 12 oz. soil jars will be baked in an oven to remove any residual VOCs that may be present. Prior to sample collection, approximately 100 mL of purge and trap grade methanol will be introduced into each sample jar. If necessary, a larger volume of methanol will be used. Caps will be added to the sample jars. The methanol volume will be recorded in the field notebook. The 12 oz. sample jars (cap on) containing methanol will be pre-weighed and numbered, and weights will be recorded in a field notebook. The sample jars will then be stored on ice or refrigerated until use.
2. Following extraction of the fractured rock interval from the drilling core barrel (depending on the nature of the fracture use of a rock saw may or may not be required), selected fracture face material (i.e. natural fracture rock chips) will be chipped away from the fracture face and placed in a 12 oz. clear wide mouth sample jar or collected in a stainless steel bowl and immediately transferred to a sample jar. Approximately 50 - 100 grams of the fractured rock material will be collected. The sample container will be capped. The sample will be weighed to determine the weight of the rock chips/fragments. Final weight will be recorded in the field notebook. The sample is allowed to bathe in the methanol for a period of 10-12 hours.
3. A disposable pipette will be used to collect an aliquot of methanol from the wide mouth sample jar. Transfer the methanol to a 2 mL amber vial and cap. Record the appropriate sample identification information on the sample label and attach the label to the vial. Submit the sample to the laboratory for VOC analysis.
4. Sample Analysis - Samples will be analyzed by purge and trap analysis using USEPA Method 5030 procedures developed for high concentration soils. Sample collection data along with the analytical results from the laboratory will be used to determine total mass of target compounds present in the fracture zone. Detection levels will be approximately 5 µg/core sample for target VOCs reported at the on-site laboratory. The following calculation will be used to determine total mass of a detected target compound:

Mass of Compound (µg) = (A * B * C)/D

A = Concentration of Aqueous Analysis in µg/L

B = Purge and trap purge volume in L

C = Volume of methanol used during sample collection in mL

D = Volume of methanol extract used during analysis in mL

Detection limit example:

5 µg = (1 µg/L * .005 L * 100 mL)/.1 mL

10.0 ATA AND RECORDS MANAGEMENT

A record of general field activities, field conditions, weather personnel, and sample locations, will be kept in the task field book, in accordance with the site Sampling and Analysis Plan (SAP) and the site Quality Assurance Project Plan (QAPP).

A rock core log record (see SAP) will be used for each boring location to log the rock core and data such as; date, boring ID, overburden thickness, total depth, and other details.

Sample collection information will be recorded in the field logbook. The sample collection information will be used in calculations of volumes of VOCs present in the rock core. The concentration of analytes present in the methanol will be reported (based on a 100 µL extract purge and a dilution factor of 50). The total amount and concentration of target compounds can then be calculated.

Total amount (µg) = Concentration in methanol µg/L * Liters of methanol used during extraction

Total Concentration (µg/g) = Total amount (µg) / mass of rock (g)

An electronic record of GPS data (if used) will be retained in the project file to document the final bedrock borehole locations.

Photographs of a typical rock core fractures and rock chip samples should be included in the project file.

11.0 QUALITY COPNTROL/QUALITY ASSURANCE

Quality control for this MERC sampling method involves the collection of field quality control samples including field duplicated, matrix spike/matrix spike duplicate samples, trip blank samples, equipment blank samples, and temperature blank samples. Frequency of collection or QC samples will be specified in the SAP.

SOP No. S-19

Wood Environment & Infrastructure Solutions, Inc.

STANDARD OPERATING PROCEDURE

CONCRETE CHIP SAMPLING

wood.

STANDARD OPERATING PROCEDURE

CONCRETE CHIP SAMPLING

1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the methods to be used for collection of porous surface samples for the analysis of non-volatile analytes. The equipment may include hammer drill, drill bits, masonry hammer, lump hammer, cold chisels, or any other type of equipment used during field activities.

A hammer drill can be used to generate a finely ground powder from hard surfaces and most soft surfaces which can be collected, extracted, and analyzed for non-volatile analytes (i.e., PCB, PCDD, metals, cyanide, etc.). Detection limits are analyte specific. Sample size should be determined based upon the amount of sample requested by the analytical laboratory. A typical sample area is one square foot but can be adjusted based upon field observations or sample volume required.

A sample depth profile can be obtained by vacuuming the sample hole between depth intervals. The hammer drill bit can then be advanced through the next depth interval and the chip sample collected from that interval. Depth profiling is typically limited by the thickness of the material sampled (i.e., concrete slab thickness).

Using a hand hammer and chisel or sharp knife, soft surfaces can be sampled if a hammer drill does not produce sufficient ground powder for sample collection.

Hard porous surfaces typically include concrete, brick, asphalt, cement, sandstone, limestone, and unglazed ceramics. Soft porous surfaces include wood, wall plasterboard, low density plastics, rubber, and caulking.

2.0 RESPONSIBILITIES

It is the primary responsibility of the project Field Operations Lead and field samplers to assure that the proper sampling procedures are followed and that all field data records are completed.

It is the responsibility of the project safety officer to draft and enforce safety measures which

provide the best protection for all persons involved directly with sampling.

It is the responsibility of any subcontractors (i.e., drilling contractors) to follow the proper designated sampling procedures that are stated in their contracts and outlined in the Site Safety and Health Plan (SSHP).

It is the responsibility of all personnel involved with sample collection to maintain a clean working environment and to ensure that all procedures are followed.

4.0 HEALTH AND SAFETY WARNINGS

This SOP does not present a hazard analysis specifically associated with this procedure and cannot address all hazards associated with sampling activities. Health and safety details such as activity hazard analysis should be referenced in a Site Safety and Health Plan (SSHP). The following are general heath and safety guidelines associated with any drilling activity conducted at an environmental investigation site.

Hammer drilling involves the use of a powerful hand-held electric drill. Potential hazards include but are not limited to pinching, crushing, piercing, repetitive motion, and noise hazards. Appropriate personal protective equipment (PPE) should be used to protect against these and other general site hazards.

5.0 PERSONNEL QUALIFICATIONS

Personnel using this SOP should be trained to the appropriate degree in hazardous waste site safety procedures as required by OSHA regulations.

6.0 PROCEDURES

Concrete Chip Sampling

Concrete chip sampling will require the following equipment:

- Electric hammer drill with 1-inch and 3/8-inch diameter drill bits
- Two 50-ft long electrical extension cords
- Various hand hammers (i.e., masonry, lump)
- Cold chisels
- Utility Knife

- Stainless steel spatulas or scoopulas
- Vacuum trap and vacuum pump
- Various brushes and stainless steel dustpan
- Shop Vacuum with HEPA filtration
- Sample containers with labels – laboratory provided
- Chain of Custody (COC) form - laboratory provided

Sampling equipment, especially the components that come into contact with the sample, must be decontaminated prior to initiating sampling activities and between each sample location. Decontamination procedures in SOP S-3 Decontamination of Field Equipment should be followed (see the site Sampling and Analysis Plan [SAP]).

Hard Porous Material Chip Sampling

1. Identify the area and depth to be sampled.
2. Ensure that the electric hammer drill is not plugged into the electrical outlet.
3. Install the one inch diameter, carbide tipped, drill bit into the electric hammer drill.
4. Plug in the hammer drill to the electrical supply.
5. Place the bit onto the area to be sampled and begin drilling.
6. When the sample depth has been attained, stop drilling and move to the adjacent area of the first hole. Continue drilling. This process can be repeated until sufficient sample volume has been attained.
7. Once sufficient sample volume has been attained, unplug the hammer drill from the electrical supply and move away from the sampling location.
8. Using the appropriate tool, transfer the chip sample to the appropriate sample containers. Label the sample containers with the sample identification, date and time collected, and the analytical tests required.
9. Document the sample in the appropriate field collection record (see SAP) and the field activities logbook.
10. Add the sample to the COC.

Soft Porous Material Chip Sampling

1. The procedures for hard porous material sampling may be applicable to certain soft porous materials.
2. Hand hammers and chisels can be used if the electric hammer drill does not provide the soft porous sample chips needed. Follow steps 5 through 10 above.

For soft porous material such as caulking and rubber, a representative sample can be obtained using a sharp utility knife.

7.0 QUALITY CONTROL/QUALITY ASSURANCE

Quality control for this sampling method involves the collection of field quality control samples including field duplicated, matrix spike/matrix spike duplicate samples, and equipment blank samples. Frequency of collection or QC samples will be specified in the SAP.

8.0 REFERENCES

USEPA, 1994. "Chip, Wipe, and Sweep Sampling"; SOP # 2011; November 16, 1994; Revision 0

USEPA Region 1, 2011. "Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)"; EIASOP_POROUSSAMPLING; May 4, 2011; Revision 4.

Appendix B

Field Data Records

FIELD ACTIVITY DAILY LOG

DAILY LOG	DATE			
	NO.			
	SHEET		OF	

Project Name:	**Project No.**

Installation/Investigation Area:

Description of daily activities and events:

List Samples Collected:

Visitors on Site:	**Deviation from plans:**
Weather conditions:	**Important telephone calls / photos taken:**

Personnel on Site:

Name/Signature:	**Date:**
QA/QC'd by:	**Date:**

FIELD INSTRUMENTATION CALIBRATION RECORD

PROJECT _____ DATE _____ TIME _____

CREW ID OR TASK ID _____ JOB NUMBER _____

SAMPLER SIGNATURE _____ CHECKED BY _____

EQUIPMENT CALIBRATION		INITIAL CALIBRATION		SECONDARY CALIBRATION (see note 3)		
MANF & MODEL NO._____		STANDARD VALUE	METER VALUE	STANDARD VALUE	METER VALUE	ACCEPTANCE CRITERIA **
UNIT ID NO._____						
pH	units					+/- 10% of standard
Redox	+/- mV					see note 1
Conductivity	mS/cm					+/- 10% of standard
DO	mg/L *					+/- 10% of standard
Thermometer Temperature	deg. C					+/- 2.0 deg. C
TURBIDITY						
METER TYPE_____	NTU (low)					within 0.5 NTU of
MODEL NO._____						the standard
UNIT ID NO._____	NTU (high)					+/- 10% of standard
PHOTOIONIZATION	Background					
METER TYPE_____	ppmv					within 5 ppmv of Zero
MODEL NO._____						
UNIT ID NO._____	Span Gas ppmv					+/- 10% of standard
OTHER METER TYPE_____						
MODEL NO._____						
UNIT ID NO._____						see note 2

Check One

☐ Equipment calibrated within the Acceptance Criteria specified for each of the parameters listed above.

☐ Equipment (not) calibrated within the Acceptance Criteria specified for each of the parameters listed above (see notes below).

MATERIALS RECORD

Deionized Water Source: _____

PID SPAN Gas: Lot _____

PID Zero Gas: Lot _____

Other : _____

Source and Lot Number

pH _____

ORP _____

Conductivity _____

Turbidity _____

Other _____

NOTES:

* = Indicate in notes section what was used as the DO standard (i.e., based on saturation at room temperature)

** = If the meter reading is not within acceptance criteria, clean or replace probe and re-calibrate, or use a different meter if available. If project requirements necessitate use of the instrument, clearly document on all data sheets and log book entries that the parameter was not calibrated to the acceptance criteria.

1 = meter must read within specified range of the Zobell solution.

2 = specify acceptance criteria in the Notes section

3 = secondary calibration to be completed should instrument drift be suspected during field day

wood.

GROUNDWATER/ PORE WATER GRAB SAMPLING RECORD

wood.
511 Congress Street, Portland Maine 04101

PROJECT NAME		SAMPLE LOCATION	DATE
PROJECT NUMBER		START TIME	END TIME
SAMPLE ID	SAMPLE TIME	SITE NAME/NUMBER	PAGE OF

SAMPLE TYPE ☐ GRAB ☐ WELL/PIEZOMETER ☐ GEOPROBE ☐ PORE WATER ☐ OUTFALL ☐ OTHER _____

WELL DIAMETER (INCHES) ☐ 1 ☐ 2 ☐ 4 ☐ 6 ☐ 8 ☐ OTHER _____

TUBING ID (INCHES) ☐ 1/8 ☐ 1/4 ☐ 3/8 ☐ 1/2 ☐ 5/8 ☐ OTHER _____

MEASUREMENT POINT (MP) ☐ TOP OF RISER (TOR) ☐ TOP OF CASING (TOC) ☐ OTHER _____

WELL INTEGRITY

	YES	NO	N/A
CAP			
CASING			
LOCKED			
COLLAR			

INITIAL DTW (BMP)	FT	**FINAL DTW (BMP)**	FT	**PROT. CASING STICKUP (AGS)**	FT	**TOC/TOR DIFFERENCE**	FT
WELL DEPTH (BMP)	FT	**SCREEN LENGTH**	FT	**PID AMBIENT AIR**	PPM	**REFILL TIMER SETTING**	SEC
WATER COLUMN	FT	**DRAWDOWN VOLUME** (initial DTW- final DTW X well diam. squared X 0.041)	GAL	**PID WELL MOUTH**	PPM	**DISCHARGE TIMER SETTING**	SEC
CALCULATED GAL/VOL (column X well diameter squared X 0.041)	GAL	**TOTAL VOL. PURGED** (mL per minute X total minutes X 0.00026 gal/mL)	GAL	**DRAWDOWN/ TOTAL PURGED**		**PRESSURE TO PUMP**	PSI

FIELD PARAMETERS

TIME	DTW (FT)	PURGE RATE (mL/min)	TEMP. (°C)	SP. CONDUCTANCE (mS/cm)	DISS. O_2 (mg/L)	pH (units)	REDOX (mv)	TURBIDITY (ntu)	PUMP INTAKE DEPTH (ft)	COMMENTS
	BEGIN PURGING									

SAMPLE OBSERVATIONS: CLEAR _____ COLORED _____ CLOUDY _____ TURBID _____ ODOR _____ OTHER (see notes) _____

EQUIPMENT DOCUMENTATION

TYPE OF PUMP	DECON FLUIDS USED	TUBING/PUMP/BLADDER MATERIALS		EQUIPMENT USED
☐ PERISTALTIC	☐ LIQUINOX	☐ SILICON TUBING	☐ S. STEEL PUMP MATERIAL	☐ WATER LEVEL METER
☐ SUBMERSIBLE	☐ DEIONIZED WATER	☐ TEFLON TUBING	☐ PVC PUMP MATERIAL	☐ PID _____
☐ BLADDER _____	☐ POTABLE WATER	☐ TEFLON LINED TUBING	☐ GEOPROBE SCREEN	☐ WQ METER _____
☐ WATTERA	☐ NITRIC ACID	☐ HDPE TUBING	☐ TEFLON BLADDER	☐ TURB. METER _____
☐ OTHER _____	☐ HEXANE	☐ LDPE TUBING	☐ OTHER _____	☐ PUMP _____
☐ OTHER _____	☐ METHANOL	☐ OTHER _____	☐ OTHER _____	☐ OTHER _____
	☐ OTHER _____	☐ OTHER _____	☐ OTHER _____	☐ FILTERS NO. ___ TYPE _____

ANALYTICAL PARAMETERS

	PARAMETER	METHOD NUMBER	PRESERVATION METHOD	VOLUME REQUIRED	SAMPLE COLLECTED	QC COLLECTED	SAMPLE BOTTLE ID NUMBERS
☐							
☐							
☐							
☐							
☐							
☐							
☐							

NOTES

SKETCH

PURGE OBSERVATIONS

	YES	NO	
PURGE WATER CONTAINERIZED	☐	☐	NUMBER OF GALLONS GENERATED _____
NO-PURGE METHOD UTILIZED	☐	☐	If yes, purged approximately 1 standing volume prior to sampling or _____mL for this sample location.

Sampler Signature: Print Name:

Checked By: Date:

EQUIPMENT BLANK SAMPLING RECORD

wood.

PROJECT NAME	SAMPLE LOCATION	PROJECT NO

Rinsate Blank Sample I.D.:	
Date/Time:	
DI Water Source:	
Equipment Used:	

Sample I.D.s associated with above Rinsate Blank	Comment

Created By: BPW
Checked By:

SOIL BORING LOG

wood. 511 Congress Street, Portland Maine 04101	Project Name:		Boring ID:
	Project Location:		Page No.
	Project No.:	Client:	of:
Boring Location:	Refusal Depth:	Total Depth:	Bore Hole ID/OD:
Weather:	Soil Drilled:	Method:	Casing Size:
Subcontractor:	P.I.D (eV):	Protection Level:	Sampler:
Driller:	Date Started:	Date Completed:	Sampler ID/OD:
Rig Type/Model:	Logged By:	Checked By:	Hammer Wt/Fall:
Reference Elevation:	Water Level:	Time:	Hammer Type:

Sample Information					Monitoring						
Depth (feet bgs) 0.0	Sample Number	Penetration/ Recovery (feet)	SPT Blows/6"	N Value	PID Field Scan	PID Headspace	Lab Tests Performed	Lab Sample ID	Sample Description and Classification	USCS Group Symbol	Remarks

NOTES:

SURFACE SOIL SAMPLING RECORD

wood.

511 Congress Street, Suite 200, Portland Maine 04101

PROJECT NAME	
PROJECT NUMBER	
SAMPLE ID	**SAMPLE TIME**

SAMPLE LOCATION	**DATE**
START TIME	**END TIME**
SITE NAME/NUMBER	**PAGE** OF

SAMPLE INFORMATION

TYPE OF SAMPLE

- ☐ DISCRETE
- ☐ COMPOSITE

SAMPLE INTERVAL:

TOP _____

BOTTOM _____

COLLECTION EQUIPMENT

- ☐ HAND AUGER/CORER
- ☐ S.S. SPLIT BARREL
- ☐ ALUMINIUM PAN
- ☐ S.S. SHOVEL
- ☐ HAND SPOON/SPATULA
- ☐ S.S. BUCKET
- ☐ OTHER _____

DECON FLUIDS USED

- ☐ LIQUINOX
- ☐ DEIONIZED WATER
- ☐ POTABLE WATER
- ☐ NITRIC ACID
- ☐ HEXANE
- ☐ METHANOL
- ☐ DI WATER N_2 PURGE
- ☐ OTHER

QC SAMPLES

- ☐ DUPLICATE _____
- ☐ EQ BLK _____

MS/MSD:

- ☐ YES
- ☐ NO

TYPE OF MATERIAL:

- ☐ ORGANIC
- ☐ SAND
- ☐ GRAVEL
- ☐ CLAY
- ☐ FILL
- ☐ OTHER_____

SAMPLE OBSERVATIONS

ODOR _____

COLOR _____

OTHER _____

FIELD SKETCH SHOWN/ATTACHED

- ☐ YES
- ☐ NO

ANALYTICAL PARAMETERS

	PARAMETER	METHOD NUMBER	PRESERVATION METHOD	VOLUME REQUIRED	SAMPLE COLLECTED	QC COLLECTED	SAMPLE BOTTLE ID NUMBERS
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							

NOTES

SKETCH

Sampler Signature: Print Name:

Checked By: Date:

WOOD ENVIRONMENT & INFRASTRUCTURE SOLUTIONS PAGE ___ OF _____

FIELD DATA RECORD - SURFACE WATER/ SEDIMENT SAMPLING

PROJECT _____ JOB NUMBER _____ DATE _____

FIELD SAMPLE NUMBER _____ ACTIVITY TIME START _____ END _____ Sample Time _____

QC SAMPLES COLLECTED _____

SURFACE WATER DATA

WATER DEPTH AT LOCATION _____ FT
SPEC. COND _____ mS/CM
DEPTH OF SAMPLE FROM SURFACE _____ FT
D.O. _____ PPM
TEMPERATURE _____ DEG C
SALINITY _____ PPM
TURBIDITY _____ NTUS
ORP _____ mV
PH _____ UNITS
Associated field Dup _____
Equip BLK _____
MS/MSD: Yes

EQUIPMENT USED
- ☐ BEAKER/Bottle
- ☐ PACS BOMB
- ☐ PERISTALTIC PUMP
- ☐ FILTER/ NUMBER _____
- ☐ OTHER- _____

TYPE OF SURFACE WATER
- ☐ STREAM/ RIVER
- ☐ LAKE/ POND
- ☐ SEEP
- ☐ MARSH
- ☐ OTHER _____

DECON FLUIDS USED:
- ☐ DI WATER
- ☐ POTABLE WATER
- ☐ _____

SEDIMENT DATA

DEPTH OF SEDIMENT _____

TYPE OF SAMPLE
- ☐ DISCRETE
- ☐ COMPOSITE

SAMPLE OBSERVATIONS
- ODOR _____
- COLOR _____

TYPE OF SEDIMENT:
- ☐ ORGANIC
- ☐ SAND
- ☐ GRAVEL
- ☐ CLAY
- ☐ TALCOSE_____
- ☐ OTHER_____

EQUIPMENT FOR COLLECTION
- ☐ HAND CORER
- ☐ S.S. SPOON
- ☐ Shovel-Trowel
- ☐ DREDGE
- ☐ OTHER _____

DECON FLUIDS USED
- ☐ DI WATER
- ☐ POTABLE WATER
- ☐ LIQUINOX
- ☐ OTHER _____

Associated field Dup _____ Equip BLK _____ MS/MSD: Yes

ANALYTICAL PARAMETERS

WATER

	METHOD NUMBER	FILTERED	PRESERVATION METHOD	VOLUME REQUIRED	Notes:
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					

ANALYTICAL PARAMETERS

SOIL

	METHOD NUMBER		PRESERVATION METHOD	VOLUME REQUIRED	Notes:
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					
☐ _____					

NOTES

SIGNATURE: _____

RECEIVED BY: _____

Tailgate Safety Meeting Report

Check One:

☐ Initial Kickoff Safety Meeting ☐ Regular/Daily Tailgate Safety Meeting ☐ Unscheduled Tailgate Safety Meeting

Date: _____ Site: _____

Site Manager: _____ Site Health and Safety Officer: _____
Print *Print*

Order of Business

Topics Discussed (Check all that apply – Boxed bold items to be covered daily)

☐ **Scope of Work**	☐ Hazard Analysis of Work Tasks (chemical, physical, biological and energy health hazard effects)
☐ **Anticipated Weather (snow, high winds, rain)**	☐ Chemical Hazards and Controls
☐ **Personnel Roles and Responsibilities**	☐ Signs and symptoms of over exposure to site chemicals
☐ **Data Collection Objectives**	☐ Physical Hazards and Controls (e.g., overhead utility lines)
☐ **Safe work practices**	☐ Biological Hazards and Controls (e.g., poison ivy, spiders)
☐ **Logs, Reports, Recordkeeping**	☐ Temperature Extremes (heat or cold stress symptoms and controls)
☐ Site History/Site Layout	☐ Engineering Controls
☐ Site Control (visitor access, buddy system, work zones, security, communications)	☐ Monitoring Instruments and Personal Monitoring, Action Levels
☐ Training/Permit Requirements	☐ Perimeter Monitoring - Type and Frequency
☐ Applicable SOPs (e.g., Hearing Conservation Program, Safe Driving, etc.)	☐ Near Misses/Hazard ID including worker suggestions to correct and work practices to avoid similar occurrences
☐ PPE Required/PPE Used	☐ Incident Reporting Procedures
☐ Define PPE Levels, Donning, Doffing Procedures	☐ Hazardous Materials Spill Procedures
☐ Decontamination Procedures for Personnel and Equipment	☐ Medical Emergency Procedures (e.g., exposure control precautions, location of first aid kits, etc.)
☐ Sanitation and Illumination	☐ Route to Hospital and Medical Care Provider Visit Guidelines
☐ Medical Surveillance Requirements	☐

Safety Suggestions by Site Workers: ☐ None Provided ☐ Input Given (record in field below)

Action Taken on Previous Suggestions: ☐ None Needed ☐ Actions (record in field below)

Injuries/Incidents/Personnel Changes since last meeting: ☐ None ☐ Occurred (record in field below)

Tailgate Safety Meeting Report

Observations of unsafe work practices/conditions that have developed since previous meeting:

Location of (or changes in the locations of) evacuation routes/safe refuge areas:

☐ Applicable Procedures (AHA, JHA, SWP):

☐ Field Level Risk Assessment Completed (FLRA) (e.g. new hazards identified due to site or equipment conditions):

Attendee signatures below indicate acknowledgment of the information and willingness to abide by the procedures discussed during this safety meeting

Name (Print)	Company	Signature

Meeting Conducted by: _____ Title: _____

Print Name

Signature: _____ Time: _____

PROJECT SITE
OCCUPATIONAL HEALTH & SAFETY INSPECTION CHECK LIST

DATE:	PROJECT LOCATION:	NAME (S) OF PERSON/PEOPLE CONDUCTING INSPECTION:
PROJECT NUMBER:	PROJECT MANAGER:	PROJECT NAME:
SITE ACTIVITIES:	WEATHER:	PERSONNEL PRESENT (AMEC, CLIENT, & CONTRACTORS):

This checklist documents AEI & Subcontractor safety compliance at the project.
Please check (ö) the appropriate box next to the specific item.

"Y" **Indicates compliance.**
"N" **Indicates non-compliance and requires immediate correction.**
"NA" **Indicates that the item is not applicable at the project.**
"CA" **Corrective action – Initials of responsible person to complete.**

	Planning and Documentation	Y	N	N/A	CA
1	Project Specific Job Hazard Assessment completed	☐	☐	☐	
2	Site Specific Safety Plan Available and signed	☐	☐	☐	
3	Project safety program for subcontractors submitted	☐	☐	☐	
4	HAZCOM/WHMIS program provided	☐	☐	☐	
5	MSDS available	☐	☐	☐	
6	Tailgate/Tool Box safety meetings held	☐	☐	☐	
7	Project orientation provided	☐	☐	☐	
8	Project Specific Safety training provided where necessary	☐	☐	☐	
9	First-aid supplies available	☐	☐	☐	
10	Qualified first aid person on project	☐	☐	☐	
11	Safety bulletins, rules, regulations, etc. posted	☐	☐	☐	
12	Emergency telephone numbers posted	☐	☐	☐	
13	Communication system in place	☐	☐	☐	
14	Signs posted where necessary	☐	☐	☐	
	General Safety	**Y**	**N**	**N/A**	**CA**
15	Slip, Trip & Fall hazards identified and cleared	☐	☐	☐	
16	Utility mark out completed	☐	☐	☐	
17	Overhead hazards identified	☐	☐	☐	
18	Safety Zones established (Exclusion, Contamination Reduction, Support)	☐	☐	☐	
19	Decontamination procedure/area established	☐	☐	☐	
20	Confined space procedures followed	☐	☐	☐	
21	Adequate ventilation in work areas	☐	☐	☐	
22	Adequate lighting provided and maintained in work areas	☐	☐	☐	
23	Sharp objects properly disposed of or protected	☐	☐	☐	
24	Proper storage of tools and materials	☐	☐	☐	
25	Accumulation of contaminated debris within acceptable levels	☐	☐	☐	
26	Adequate trash containers provided	☐	☐	☐	
27	Adequate number of toilets and washing facilities	☐	☐	☐	

	Personal Protective Equipment	Y	N	N/A	CA
28	Hardhats worn by workers	☐	☐	☐	
29	Safety glasses or protective eyewear used when required	☐	☐	☐	
30	Appropriate respirators used when required	☐	☐	☐	
31	Proper work shoes worn by all employees	☐	☐	☐	
32	Appropriate hearing protection used when required	☐	☐	☐	
33	Safety vests worn when required	☐	☐	☐	
34	Proper protective clothing used when required	☐	☐	☐	
35	Personal Flotation Devices (PFD) utilized when required	☐	☐	☐	

	Fire Protection and Prevention	Y	N	N/A	CA
36	Fire suppression equipment available and inspected routinely	☐	☐	☐	
37	Flammable and combustible materials stored properly	☐	☐	☐	
38	Flammable liquid stored in approved safety cans	☐	☐	☐	
39	Safety cans have self closing lids and flame arresters	☐	☐	☐	
40	Combustible waste materials routinely disposed of	☐	☐	☐	
41	Flammable containers properly labeled	☐	☐	☐	

	Tools: Hand and Power	Y	N	N/A	CA
42	Proper tool used for job	☐	☐	☐	
43	Hand tools in good condition and free of visible defects	☐	☐	☐	
44	Guards in place	☐	☐	☐	
45	Tool handles not broken	☐	☐	☐	
46	Electric tools double insulated or properly grounded	☐	☐	☐	
47	Power cords on electric tools in safe working condition	☐	☐	☐	
48	Powder actuated tools: operators certified	☐	☐	☐	
49	All belts, chains, sprockets and pulleys properly guarded	☐	☐	☐	
50	Power finishing machines equipped with dead man's switch	☐	☐	☐	

	Electrical	Y	N	N/A	CA
51	GFCI or assured grounding in use	☐	☐	☐	
52	Extension cords are approved three wire construction grade	☐	☐	☐	
53	Extension cords free of visible defects	☐	☐	☐	
54	Extension cords not running through water	☐	☐	☐	
55	Extension cords strung to avoid damage	☐	☐	☐	
56	Temporary lighting properly guarded	☐	☐	☐	
57	Temporary lighting properly suspended	☐	☐	☐	
58	All live circuits and panels clearly posted	☐	☐	☐	
59	Live panels secured to prevent unauthorized access	☐	☐	☐	
60	Only qualified persons working on live circuits and panels	☐	☐	☐	
	Fall Protection	**Y**	**N**	**N/A**	**CA**
61	Excavations properly guarded to prevent fall	☐	☐	☐	
62	Workers by excavation openings utilized fall protection if deeper than 6'	☐	☐	☐	
63	Full body harnesses used as fall protection at unprotected edges greater than 6'	☐	☐	☐	
64	Harnesses are properly worn by worker	☐	☐	☐	
65	Lanyard of proper length to limit fall to less than six feet	☐	☐	☐	
66	Lanyards secured to proper anchorage	☐	☐	☐	
67	Lifelines secured to proper independent anchorage	☐	☐	☐	
68	Controlled access zone warning lines in place	☐	☐	☐	

	Heavy Equipment (Backhoe,Excavator, Drill Rig, Loader)	Y	N	N/A	CA
69	Permits, inspections and licenses in order and valid	☐	☐	☐	
70	Daily inspection of equipment performed	☐	☐	☐	
71	Backup alarm operational	☐	☐	☐	
72	Signal person provided	☐	☐	☐	
73	Clearance to power lines is adequate (20')	☐	☐	☐	
74	Backhoe outriggers fully extended and supported during operation	☐	☐	☐	
75	Boom down prior to drill rig movement	☐	☐	☐	
76	Personnel properly positioned	☐	☐	☐	

	Ladders	Y	N	N/A	CA
77	Ladders are free of visible defects	☐	☐	☐	
78	Ladders proper height for work	☐	☐	☐	
79	Workers do not overextend reach of ladders	☐	☐	☐	
80	Ladders erected on solid level surface	☐	☐	☐	
81	Nonconductive ladder is used when necessary	☐	☐	☐	
82	A-frame ladders used in open position	☐	☐	☐	
83	Workers do not use top two steps of A-frame ladders	☐	☐	☐	
84	Workers do not climb back of A-frame ladders	☐	☐	☐	
85	Straight ladders secured	☐	☐	☐	
86	Straight ladders extend 36 inches above landing	☐	☐	☐	
87	Straight ladders pitched at 1 to 4 ratios	☐	☐	☐	
88	No skid feet provided on straight ladders	☐	☐	☐	
	Public Liability	**Y**	**N**	**N/A**	**CA**
89	Fencing provided where necessary	☐	☐	☐	
90	Warning signs posted where necessary	☐	☐	☐	
91	Flag persons used to direct pedestrian and vehicle traffic if needed	☐	☐	☐	
	Life Safety	**Y**	**N**	**N/A**	**CA**
92	Evacuation plans posted	☐	☐	☐	
93	Paths of emergency egress kept clear	☐	☐	☐	
94	Rescue equipment and team available	☐	☐	☐	

	Excavation	Y	N	N/A	CA
95	Sheeting, shoring and bracing in place (excavation greater than 4')	☐	☐	☐	
96	Sloping and bracing where necessary (excavation greater than 4')	☐	☐	☐	
97	Ingress and egress provided (excavation greater than 4')	☐	☐	☐	
98	Guardrails in place (excavation greater than 4')	☐	☐	☐	
99	Spoils two feet from excavation (excavation greater than 4')	☐	☐	☐	

NOTE: Based on the results of this inspection, all causing, exposing and contractors responsible for correcting deficiencies and non-compliance will be contacted in writing to perform necessary corrective actions.

COMMENTS/ NOTES:

Use back or additional pages for comments and explanations.

Appendix C

Laboratory Standard Operating Procedures

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	Minor changes rewording of sec 7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practices and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 4 - added list of waste streams generated and location of satellites. Clarified RT window studies. Added reference to MI Sop. Removed Grand Mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying Calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement can not be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10
LAD 05/11/11 12	Removed Tekmar 2000 and 2016 throughout. Sect. 7.3.1 - Removed ~~5970~~ 5970 GC/MS instrument type. Sect. 7.4.7 - Added RRT information. Sect. 8.1 - Added S.C. marginal exceedence criteria. Sect. 9 - Added MDL, LOD and LOQ Criteria. Updated Figures.	LAD	05/11	05/11
13	Sect. 5 - Changed Cal mix and ICV Std. Exp. from 7 to 14 days. Sect. 6 - Add Sample preservation info. Sect. 7.4.4.1 - Added S.C. exemption from 2nd source cal. Sect. 7.5.1 - Added Extras mix to LCS. Sect. 7.6.12 - clarified Nothing why samples need to be reanalyzed. Sect. 8.1 - Added 10% rule for LCS, ICV and MS/D. Sect. 9 - Added LOD/LOQ definitions. Table 1 - Reworded CA	LAD	03/12	03/12
14	for ICV. Sect. 1 and 7 - Removed Quickform references and added reporting from KIMS. Sect. 7 - Removed Soil 200 ug/kg level and added 80.0% level. Sect. 8 - Added additional marginal exceedance information. Throughout - Fixed typos and made minor edits.	LAD	04/13	04/13
15	Sect. 4 - Removed 5890, 5972 and Tekmar references. (5970 too). Sect. 10 - Updated and added references. Table 3 - Added DoD QSM Ver. 5.0 QC requirements. Renumbered Tables 3, 4, 5.	LAD	04/14	04/14

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
16	Sect. 1 – Added sand to m. blk def. Sect. 5 – Separated cal std into VOA gases and non-gases, clarified VOA gases exp. date, combined Surr & IS std, added sand. Sect. 6 – Added Acrolein and Acrylonitrile preservation. Sect. 7 – Added poor purgers % RSD calibration info, added CCV criteria, added use of sand in soil Blank and LCS's, updated cal spike prep. Sect 8 – Added additional LCS criteria. Updated Figures 1 – 3.	LAD	07/16	07/16
17	Sect. 7 - Update BFB Method naming convention, Update Soil calibration levels, Added % Error calculation. Sect. 9. Added LLOQ reference to LOQ verification.	LAD	03/17	03/17
18	Sect 1, 8, 9 and/or Table 1 - Added LLOQ definition, and LLOQ verification criteria, clarified PQL, LOQ and LLOQ. Added % Error criteria Sect. 7 - Corrected RSE requirement	LAD	06/17	06/17
19	Sect. 5 - Changed IS and Surrogate Standard expiration date from 14 to 30 days. Removed references for 1-Chlorohexane. Removed Table 2 - DoD/Isom 4.2 QC criteria. Updated logbook example. Updated references	LAD	10/18	10/18
20	Sect. 4 - Updated column, data acquisition and processing systems. Sect. 5 - Updated final concentrations and expiration dates for non-gas stds. Sect. 7 - Updated calib. high pt. conc.	LAD	01/20	01/20

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-20,** titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-20**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

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TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices

common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil

samples must be decanted, and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire concentrator must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph (GC): Hewlett Packard 6890.

4.2 Mass Spectrometer (MS): HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Column: RTX-VMS, 30 meter, 0.25 mm ID or equivalent.

4.5 Purge and Trap: Archon or Centurion auto samplers, and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Agilent Chemstation or equivalent.

4.9 Data System: KIMS is used for processing data. The Target software is used for generating forms.

5.0 REAGENTS AND STANDARDS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”. After ampulated standards are cranked open, the standard is transferred to a screw top vial and stored in a freezer.

5.3.1 The expiration date for all standards except volatile gases is six months from date of opening the ampule.

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix (without gases) – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

1,2-Dibromo-3-chloropropane
1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Butanone
2-Chloroethylvinyl ether
2-Chlorotoluene
2-Hexanone
4-Chlorotoluene
4-Methyl-2-pentanone
Acetone
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Carbon disulfide
Carbon Tetrachloride
Chlorobenzene
Chloroform
cis-1,2-Dichloroethene
cis-1,3-Dichloropropene
Cyclohexane
Dibromochloromethane
Dibromomethane
Ethylbenzene
Hexachlorobutadiene
Idomethane
Isopropylbenzene
Methyl tert-butyl ether
Methylene chloride
Naphthalene
n-Butylbenzene
n-Propylbenzene
p-Isopropyltoluene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethene
Tetrahydrofuran
Toluene
trans-1,2-Dichloroethene
trans-1,3-Dichloropropene
Trichloroethene
Vinyl Acetate

5.3.2.2 Gases Calibration Mix - Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Bromomethane
Chloromethane
Dichlorodifluoromethane
Trichlorofluoromethane
Vinyl Chloride
Chloroethane

5.3.2.3 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

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Acetonitrile	Isobutyl alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl chloride	Methyl acetate
Chloroprene	Methyl methacrylate
Diethyl ether	Methyl tert-butyl ether
cis-1,4-Dichloro-2-butene	Pentachloroethane (only by special request)
trans-1,4-Dichloro-2-butene	Propionitrile
1,4-Dioxane	Tertiary-amyl methyl ether
di-Isopropyl Ether	Tertiary-butyl alcohol
Ethyl methacrylate	1,3,5-Trichlorobenzene
Ethyl tertiary-butyl ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.4 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

5.3.2.5 Surrogate/Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 30 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Internal Standards	Surrogate Standards
Pentafluorobenzene	4-Bromofluorobenzene
1,4-Difluorobenzene	1,2-Dichloroethane-D_4
Chlorobenzene-D_5	Toluene-D_8
1,4-Dichlorobenzene-D_4	Dibromofluoromethane

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 50 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

5.4 Organic Free Sand – Ottawa Sand or equivalent baked at 110 ºC overnight

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples

All aqueous samples are collected in 40 mL VOA bottles with no headspace, preserved with 1:1 HCl to a pH of <2 and stored at <6°C until analysis. Aqueous samples must be analyzed within 14 days from sample collection if preserved and within 7 days from sample collection if unpreserved.

Samples requiring Acrolein and Acrylonitrile analysis, require preservation of pH of 4-5 and cool to 0-6ºC.

6.2 Soil Samples

Soil samples arriving at the laboratory in Terra-core or Encores Soil samplers must be extruded into water or sodium bisulfate within 48 hours of sampling. Soils samples extruded into water must be frozen at -15°C ± 5°C until analysis. Soil sample extruded into sodium bisulfate must be stored at <6 °C until analysis.

Medium level soil (methanol preserved) samples are sampled into pre-weighed vials containing 5 mLs methanol. Methanol preserved soil samples must be stored at <6°C from the time of receipt at the lab until analysis.

Bulk soil samples are stored at <6 °C until analysis.

All soil/sediments must be analyzed within 14 days from sample collection.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS - Conventions for all instruments are as follows:

- Sub-Directory for data acquisition: C:\HPCHEM\1\DATA
- Tune file: BFB.U
- Method files:

For BFB Tune: VOABFBAQ.M (waters) or VOABFBSL.M (soils)
For all samples and standards: I8A05(xx)D.M
where: I = instrument ID (Each instrument is given a unique identifier).
A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

XX = the calibration number in chronological order

- Data files:
  For BFB: IB_ _ _.D

  where: I is the instrument ID
  _ _ _ is a number in chronological order from 000 to 999.

  For all other data files: I_ _ _ _.D
  Where: I is the instrument ID
  _ _ _ _ is a number in chronological order from 0000 to 9999.

  This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

Column:		RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:		150°
Run time:		8 minutes
Scan start time:		3 minutes
Scan parameters:		not to exceed 2 sec per scan
Mass range:		35-300
Number of A/D samples:		8
GC peak threshold:		1000 counts
Threshold:		10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

Purge and Trap conditions:

Parameter	Aqueous	Soil
Standby:	35°	35°
Prepurge:	0 min	0 min
Preheat Temp:	Ambient	40°
Sample Temp:	Ambient	40°
Purge:	11 min	11 min
Purge Flow Rate	~24-40 mL/min	~24-40 mL/min
Dry purge:	2-4 min	2-4 min
Desorb preheat:	245°	245°
Desorb Temp:	250°	250°
Desorb Flow Rate:	~15 mL/min	~15 mL/min
Desorb time:	2-5 min	2-5 min
Dry purge:	2-4 min	2-4 min
Bake Time:	10 min	10 min
Bake Temp:	260°	260°
Auto drain:	On	On
Bake gas by pass:	Off	Off
Valve Temp:	120°	120°
Line Temp:	120°	120°
Runs per sample:	1	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

Please refer to the Encon, Archon and Centurion Opperating manuals for more specifics on programming features.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 150 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 150 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

Notes	STD. ID	CAL. Mix 100 ug/mL	Extras Mix 100 ug/mL
AQ curve only	VSTD001	1 uL	1 uL
	VSTD005	5 uL	5 uL
SL curve only	VSTD010	10 uL	10 uL
	VSTD020	20 uL	20 uL
CCV	VSTD050	50 uL	50 uL
	VSTD100	100 uL	100 uL
	VSTD150	150 uL	150 uL

The Surrogate & Internal Standard is spiked by the autosampler. The Archon Surrogate/IS Mix is at 250 ug/ml and the instrument spikes 1 ul. The Centurion Surrogate/IS Mix is prepared at 50 ug/ml and the instrument spikes 5 ul.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Note 1: For poor purging compounds like acetone, the %RSD value may exceed the method acceptance limit of 15% but meet the acceptance criteria for the linear and quadratic calibration models. The average calibration model should still be used because this calibration model is more accurate at concentrations near the LOQ than either the linear or quadratic calibration models.

This is common for acetone but also may apply to other poor purging ketones.

In any instance the % RSD must be below 30%.

Note 2: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.4.4.2 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤15% for good performing compounds and ≤30% for poor performing compounds.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

For projects or clients requiring DoD QSM, current revision, all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.4, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM, current version, all project analytes must have $\pm$ 20%D.

For all other projects, all project analytes should have $\pm$ 30%D ($\pm$ 40%D for poor performers).

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS and Extras standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution plus 5 g baked Ottawa sand, in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

NOTE: In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water plus 5 g baked Ottawa sand, spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the 1/10th the measured amount in any sample or 1/10th the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

For projects or clients requiring DoD QSM, current version, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks

and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

NOTE: In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, "Basic Laboratory Technique", current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Archon Autosamplers

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

7.6.1.2 Centurion Autosamplers

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, it must be noted in the comments section of the injection logbook. Additional information may be needed to assure that any questions that arise during the review process can be answered.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor samples analyzed after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil

logbook. If possible, the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the autosampler or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. In this case, the Archon units may be used for the preparative step.

7.6.2 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.
Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials

back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating

the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into Kims. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.2 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. For non-DOD clients, the exceedances from the laboratory established limits or nominal limits must be less than ten percent of the client compound list. For DOD clients, all of the compounds of interest must fall within either Katahdin's statistically derived limits or the DOD QSM, current version, limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state. Additionally, the laboratory statistically derived LCS limits should fall within 70-130%.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

For projects or clients requiring DoD QSM, current version, all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.3 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.4 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be

evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260C.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, current Version.

The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all a nalytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also. % Error ≤ 30%	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances.	If the surrogate recoveries in the ICV are low but the target analytes are acceptable, narrate. If the ICV recovery is high but the sample results are <PQL, narrate. If the ICV is out but the batch LCS is in criteria, narrate.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL/LLOQ with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
MDL Studies, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-20	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Di-Isopropyl ether	45	43, 87
Ethylbezene	91	106
Ethyl methacrylate	69	41, 99
Ethyl tertiary-butyl ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl alcohol	43	41, 42
Isopropylbezene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene chloride	84	86, 49
Methyl acetate	43	74
Methyl methacrylate	69	41, 100
4-Methyl-2-pentanone	43	58, 85, 100
Methyl tert-butyl ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
n-Propylbenzene	91	120
Styrene	104	78
Tertiary-amyl methyl ether	73	55, 87, 71
Tertiary-butyl alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
Xylenes (Total)	106	91

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 5

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenzene	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butyl ether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl ether			n-Butylbenzene
Carbon disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES

GCMS-T INSTRUMENT RUNLOG **DATE/TIME OF BFB INJECTION:**

Method - Check One:	8260	8260 - 10 mL	8260 SIM	624	524
STD IDs: * BFB STD:	CAL STD:	GAS STD:	XTRAs STD:	LCS/MS STD:	
SURR STD:	IS STD:	Prep Method	Criteria	pH Paper Lot #:	KI Paper Lot #

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	5030	5035	1311	KAS	DoD	QAPP	Y/N	ANALYST	pH	TRC	COMMENTS

* Refer to page 1 for standard preparation instructions.

VOA-009 - Revision 4 - 10/17/2018

0000007

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS VOLATILES LABORATORY

REVIEWED BY/DATE:

AMP4638 Rev. PVOC/4RO Mix RLC
Ultra Scientific 6/2/16
4ST-100-1 JAP
Lot CP-1618 exp 5/31/19

AMP 4639 REVISED PVOC/4RO MIX 060616 AAB
SUPELCO 47578-U
LOT XA13795V X 04/2018

Amp 4640 ↓ 41

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-502A-R2-10X
VOC Liquid Mixture - Modified
2.0 mg/mL in Methanol
Lot: 215031197-01
Exp: Mar 24, 2018
1 mL
53 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H336 H320 H315 H311 H332 H301 H350 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Refrig (0-5 °C)
Danger
RLC 6/14/16 EPL

Amp 4642 ↓ 43

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
APP-9-048-R1-20X
Chloroprene
2.0 mg/mL in MeOH
Lot: 216031093
Exp: Mar 04, 2018
1 mL
1 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H336 H320 H315 H311 H332 H301 H351 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Freeze (<-10 °C)
Danger
Ref 06/23/16

Amp 4644 ↓ 45

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-8240C-R3-10X
Appendix IX Volatiles Mix
Varied conc. in MeOH
Lot: 214041128-01
Exp: May 18, 2018
1 mL
12 comp(s)
HIGHLY FLAMMABLE
FOR LABORATOF
H225 H336 H320 H:
H311 H332 H301 H:
P338 P360 P331 P
P262 P202 P264 P:
Storage: Refri
Danger

Amp 4646 ↓ 47 ↓ 48

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-502B-10X
Volatile Organic Cmpds - Gases
2.0 mg/mL in MeOH
Lot: 216031159
Exp: Mar 11, 2019
1 mL
6 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H330 H320 H315 H311 H332 H301 H350 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Refrig (0-5 °C)
Danger

VOA-022 – Revision 1 – 10/15/2013

QAMS589

0000013

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V0391	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 524 ES/ES	4-BFB	Amp 4413	2000	250
FINAL CONC (ug/mL): 250	1,2-DCB	4525	↓	250
FINAL VOLUME (mL): 4	Fluorobenzene	↓ 4634		250
PREP DATE: 070716				
EXPIRATION DATE: 073116				
MEOH VOLUME (uL): 3500				
MEOH LOT #: DR303				
INITIALS:				
			070716	

CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

Reviewed by/Date:

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-209
Revision History
Cover Page
Page 1

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

Prepared By: GC/MS Group Date: 7/97

Approved By:

Group Supervisor: [signature] Date: 021301

Operations Manager: [signature] Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2·12·01

General Manager: [signature] Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Format changes, added pollution prevention. Changed wording around for section 7 and updated MS spiking.	DN	2·12·01	2·12·01
02 1311	added wording to section 8 minor changes to sections 8.2, 8.3 and table 1.	LAD	4/04	4/04
03 1311	grammatical and formatting correction	LAD	04/06	04/06
04 1311	Section 5 4 changed location of pH cal - LB. 4.3 removed acid washing procedure (req. for Metals only). 1.4 added waste stream information and proper disposal. Updated Tables & Figs. Added QC table, Analyte List (Table 3), Rotary Extractor Vertical LB (Fig 2), Rotary Extractor (Fig 3) and ZHE Vessel (Fig 4). Added wording to Sect. 10 and 9. Added DOC info to Sect 8	LAD	03/07	03/07
05	Sect. 7.16 - corrected room temperature criteria. Table 2 - added method modifications for procedure of adding extraction fluid and the pressure the ZHEs are tumbled at. Updated logbook page.	LAD	11/08	11/08

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect.2 – Added more sample composition options. Sect. 7 - Added that the metals group determines % solids. Added weighing information. Updated the ZHE pressurization process. Sect. 10 - Added references. Updated figures 1 and 2.	LAD	06/12	06/12
07	Sect. 8 - Reworded MS criteria to comply with the method.	LAD	06/13	06/13
08	Added that all liquids are filtered. Updated references. Update TCLP Logbook example. Removed Rotary Extractor RPM logbook example.	LAD	07/19	07/19

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-209-08**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-209-08**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services technical personnel to extract solid matrix samples for volatile organics per EPA Method 1311, TCLP, using Zero Headspace apparatus.

1.1 Definitions

TCLP EXTRACTION BATCH: 20 or fewer samples, which are prepared together with the same method.

TCLP BLANK: An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Baked organic-free sand is used as a blank matrix. The same extraction fluid used for the samples is used for the associated TCLP blank. The blank is taken through the appropriate steps of the process.

MATRIX SPIKE (MS): Predetermined quantities of stock solutions of all TCLP compounds are added to a sample matrix and a sample matrix duplicate after filtration of the TCLP extracts and prior to sample analysis. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The purpose of the matrix spike is to monitor the performance of the analytical methods used.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the Toxicity Characteristic Leaching Procedure by EPA Method 1311. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the TCLP Method 1311 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

1.3 Safety

When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.

Always wear gloves, safety glasses and lab coat when handling the ZHE, sample or extract.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and follow appropriate procedures such as: wearing safety glasses and gloves when working with chemicals or near an instrument; not taking food or drink into the laboratory; each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Waste accumulated from the ZHE vessel is classified as organic soil waste stream “I,” which consists of leftover solids and used Borosilicate filters. The satellite for organic soil waste stream “I” is located inside the fume hood in the Volatile Organics Laboratory (room 111).

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD (taken from Method 1311)

The method in which a sample is initially handled is dependent upon the composition of the sample. There are four possibilities to consider:

- the sample has a high degree of moisture but has greater than 0.5% solids
- the sample is 100% liquid (those containing less than 0.5% dry solid material)
- the sample is 100% solids
- the sample is incompatible with the extraction fluid, such as a free product

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter and is defined as the TCLP extract.

For liquid wastes, the 100% liquid portion is defined as the TCLP extract.

For wastes containing 100% solids, the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter and is defined as the TCLP extract.

If the sample appears to be a free product/oil, the extract should be analyzed as a medium level soil.

3.0 INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical SOPs.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

4.0 APPARATUS AND MATERIALS

4.1 The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30±2 revolutions per minute (rpm) – see Figure 2. The rotation rate of each extractor is monitored before each use by counting the amount of revolutions in on minute. This is recorded the TCLP Extraction Logbook (see Figure 1). If the measured rotation rate of an extractor is outside the range 30±2 rpm, it must be taken out of service until it can be repaired. (Associated Design and Manufacturing Co. or equivalent)

4.2 ZHE Vessels - The ZHE allows for initial liquid/solid separation, extraction and final extract filtration without opening the vessel – see Figure 3. The vessels have an internal volume of 500-600 mL and are equipped to accommodate a 90-110 mm filter. (Associated Design and Manufacturing Co. Model 3745-ZHE or equivalent).

4.3 Filters - Borosilicate glass fiber containing no binder materials having an effective pore size of 0.6 to 0.8 µm. (Environmental Express Cat. #FG7590MM, size - 90 mm, pore size - 0.7 µm, or equivalent). Prefilters must not be used. Glass fiber filters are fragile and should be handled with care.

4.4 pH Meters accurate to ±0.05 units at 25°C.

4.5 60 mL Gas-tight B-D Syringe - Collection device for initial liquid phase and the final extract of the waste when using the ZHE device.

4.6 ZHE Extraction Fluid Transfer Device - A 500 mL graduated cylinder which is capable of transferring the extraction fluid without changing the nature of it. (Associated Design and Manufacturing Co. Model #3775 or equivalent)

4.7 Laboratory Balance accurate to within ±0.1 grams (all weight measurements are to be within ±0.1 grams).

4.8 Beaker or Erlenmeyer flask, glass, 500 mL.

4.9 Magnetic stirrer.

4.10 Nitrogen tank complete with gauge as appropriate.

5.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the

accuracy of the determination. The purity of all chemicals must be known or evaluated before use to minimize any laboratory contamination.

5.1 Reagent water – Laboratory grade reagent water containing no interferent. Reagent water should be monitored periodically for impurities.

5.2 Sodium hydroxide (10N), NaOH, made from ACS reagent grade.

5.3 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.4 Extraction fluid 1 – Prepared and documented in Metals Laboratory. Please refer to the current revision of Katahdin Analytical Services SOP CA-510 for further information.

NOTE: The extraction fluid should be monitored frequently for impurities. The pH must be checked and documented prior to use to ensure that these fluids are made up accurately. Documentation of pH meter calibration prior to use is to be maintained in the pH Meter Calibration Logbook (metals prep lab). If impurities are found in the extraction fluid or the pH of the fluid is not within 4.93 ± 0.05, the fluid shall be discarded and fresh extraction fluid prepared and documented.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 30 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C (±2°C) and opened only immediately prior to TCLP extraction.

6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Sample holding times for Volatile TCLP extraction and analysis are as follows:

Date of sampling to TCLP extraction: 14 days
TCLP extraction to analysis: 14 days

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Adjust the piston within the ZHE to a height that will minimize the distance it will have to move once the ZHE is charged with sample. It may be necessary to moisten the O-rings with extraction fluid to adjust the piston.

7.2 Determine if the sample is a liquid waste, if it's 100% solids, if the waste contains low solids, or if it is a free product/oil.

7.3 If the waste will obviously yield no liquid when subjected to pressure filtration, i.e., appears to be 100% solids, proceed to 7.15.

7.4 If the sample appears to contain low total solids (high degree of moisture but greater than .5% solids), the TCLP Metals group or Wet Chemistry will determine the percent solids of the sample. From this determination, approximate the amount of waste necessary so that after the liquid has been expressed there will be approximately 10 g of solid waste in the vessel and continue with step 7.7. The vessel can only be charged once.

$$\text{Weight of waste to charge ZHE} = \frac{10}{\%\ \text{solids}} \times 100$$

7.5 If the sample is less than 0.5% solids, it will be run as an aqueous sample. All aqueous samples are filtered. This filtrate is defined as the TCLP extract and is analyzed directly (See section 7.18).

7.6 If the sample appears to be an oil, extract as a medium level soil.

SAMPLE EXTRACTION WITH LOW SOLIDS

7.7 If the ZHE has been pressurized (determination of percent solids), release gas pressure on the ZHE piston, open the ZHE and force the piston back down until there is enough room for the extraction fluid.. Add the appropriate amount of extraction fluid #1 based on the weight of sample using the 500 mL graduated cylinder. If the extraction fluid was prepared on the same day as sample extraction, ensure that fluid prep information has been recorded on the bench sheet in the Volatile TCLP Extraction Logbook. Otherwise, reference the date of fluid preparation on the bench sheet. Check the ZHE to ensure that there are no leaks. Pressurize the ZHE with 5-10 psi and check again for leaks. When the pressure is at 10 psi and no leaks appear, slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the addition of extraction fluid. Stop the bleeding at the first appearance of liquid from the valve since the

appearance of liquid is an indicator of no headspace. Continue to pressurize the ZHE to 40 psi and document the pressure in the logbook.

7.8 Place the ZHE in the rotary agitation apparatus and rotate at 30±2 rpm for 18±2 hours. Record the TCLP extraction start and end time in the Volatile TCLP Extraction log. Room temperature must be maintained and documented to be at 23 ± 2°C during agitation.

7.9 After the 18±2 hour agitation period, check the pressure gauge on the ZHE to ensure there were no leaks over that time period. If the pressure within the device has been maintained, the sample in the ZHE vessel is separated into its component liquid and solid phases, as discussed in Steps 7.8 - 7.9. The liquid is carefully dispensed from the collection syringe into a VOA vial at a rate that precludes effervescence.

If the original waste contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained and the initial liquid phase are collectively defined as the TCLP extract.

If the individual phases are analyzed separately determine the volume of the individual phase (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average, as follows:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where: V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

If the individual liquid phases are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the TCLP extract and these are analyzed together.

ZHE WITH PRELIMINARY DETERMINATION OF PERCENT SOLIDS

7.10 Quantitatively transfer the entire sample (both liquid and solid phases) quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.

7.11 When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.

7.12 Attach a preweighed collection syringe to the liquid inlet/outlet valve (top flange) and open the valve. Attach the gas line to the gas inlet/outlet valve and pressurize to 1-10 psi slowly. Carefully increase the pressure to 50 psi at 10 psi increments (monitor collection syringe to prevent excessive pressure buildup which could detach or break the syringe). At each 10 psi increment, wait 2 minutes for additional liquid flow. Stop filtration when liquid flow has ceased within a 2 minute period at 50 psi.

CAUTION: Too much pressure at once can degrade the glass fiber filter and may cause premature plugging.

7.13 Reweigh the collection syringe to determine the percent solid. The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

$$\% \text{ Solids} = \frac{\text{Weight of initial sample - liquid}}{\text{Total weight of waste}} \times 100$$

The liquid phase may be analyzed immediately or stored at 4°C until time of analysis.

7.14 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended.

Proceed with step 7.15.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

ZHE FOR WASTES WITH 100% PERCENT SOLIDS

7.15 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended.

7.16 Weigh out a 10 gram sub-sample of the waste and record the weight in the Volatile TCLP Extraction Logbook (Figure 1). Quantitatively transfer the entire sample quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.

7.17 Determine the amount of extraction fluid #1 to add to the ZHE. If the waste appears to be less than 0.5 % liquid, or basically dry, use 10 g waste and 200 mL extraction fluid #1.

$$\text{Amount of extraction fluid} = \frac{20\ (\%\ \text{solids})\ (\text{weight of waste filtered})}{100}$$

7.18 Refer to Katahdin SOP CA-202, Analysis of VOAs by SW-846 Method 8260, current revision, for detailed procedure for GC/MS calibration and analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to the current revision of Katahdin SOP CA-202, Analysis of VOAs by Method 8260, for applicable quality control criteria. The following QC samples are prepared with each TCLP extraction batch:

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Blanks: 1 blank is ananlyzed per set per day. 1 ZHE vessel is loaded with 10 grams of baked sand, 200 mL extraction fluid #1, and analyzed as if it were a regular sample.

8.2 Matrix Spike - A matrix spike shall be performed for each waste type (e.g.,wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

Matrix spikes are added at a concentration equivalent to the corresponding regulatory limit, but not less than 5 times the method detection limit.

Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%\ Recovery) = 100\ (X_s - X_u)/K$$

Where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

Measured sample values are reported without correction for analytical bias (based on the matrix spike recovery).

8.2.1 Preparation of Matrix Spike: The matrix spike is prepared with the method 8260 LCS mix. Refer to the 8260 SOP CA-202, current revision for further details.

8.4 Demonstration of Capability - Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Final Update III, Method 1311, US EPA, 12/96 or current revision

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid. One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical method.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples. Remove extraction vessel from service.
	Matrix Spike	One per TCLP batch (required). One per waste type (suggested, left to discretion of client).	Refer to individual analytical method.	Refer to individual analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-209-08	METHOD 1311, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling	Leachate is drawn into a 60 mL gas tight syringe and is dispensed into VOA vials at a rate that precludes effervesence.	Method recommends the use of TEDLAR bags and or 600 mL gas tight syringes.
Procedures	Preliminary TCLP evaluations done on 10 g aliquot of waste; this subsample is also used for TCLP extraction. Extraction fluid is added to the ZHE prior to addition of sample ZHEs are tumbled at 40 ± 2 psi	Preliminary TCLP evaluations done on minimum 100 g aliquot of waste, which may not actually undergo TCLP extraction. Sample size for TCLP extraction is based on 25 g of solid in the waste subsample. Extraction fluid is added through the inlet valve. ZHEs are tumbled at 5 - 10 psi
QC - Method Blanks	Frequency of one method blank per 20 extractions or each batch.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC – Spikes	One Matrix Spike and Matrix Spike Duplicate extraction is performed for every 20 extractions.	A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.
QC – LCS		
QC – Accuracy/Precision		
QC – MDL		

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
Methyl ethyl ketone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl Chloride	0.2

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

FIGURE 1

EXAMPLE OF VOLATILE TCLP EXTRACTION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

VOLATILE TCLP EXTRACTION BENCH SHEET FOR SAMPLE SM7275-3A

Balance ID: S/N 5012 Vessel ID: E

Dry Weight Determination 100%_____ <0.5%_____ %Wet Solids_____

_____Refer to non-volatile TCLP extraction sheet, page #_____ for _____-_____

Sample description: Sludge Cake Homogeneous ✓ Non-homogeneous_____

1)	Weight Container 1 + Residue	_____g	7)	Weight of Filter	_____g
2)	Weight Container 1 + Waste	_____g	8)	Wt of Filter and Wet Solid	_____g
3)	Weight of Waste ((2) - (1))	_____g	9)	Wt of Wet Solid Phase ((8) - (7))	_____g
4)	Weight of Container 2	_____g	10)	%Wet Solids (100*(9)/(3))	_____
5)	Weight of Container 2 and Filtrate	_____g	11)	Wt of Filter + Solid Phase Dry	_____g
6)	Wt(g)/Vol(mL) of Filtrate ((5) - (4))	1	12)	Wt of Solid Phase Dry ((11) - (7))	_____g
			13)	%Dry Solids (100*(12)/(3))	_____

Analyst CR/HG Date 7/22/19 15:22

Extraction Fluid Preparation and pH Check

Refer to the Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook located in the Metals Preparation Laboratory for the Fluid #1 Preparation documentation.

Analyst JK Date 7/18/19 Extraction Fluid Batch # 8BT 1543

pH of Fluid Today 4.89 pH Criteria: 4.88 – 4.98 pH Meter ID: Orion 520A S/N 7442

Extract Use Filter Paper Lot #: 112167-9129

___ <0.5% Solids (Filter adequate sample volume) _____mL Filtered.

___ 100% Solids

10.21 g of Waste added to 200 mL of fluid (fluid volume = 20 times wt of solid sample)

___ Free Liquid present (See Equation A for amount of waste to filter)

(A) X = Desired weight of solid phase on filter * 100/ (%Wet Solid)

1)	Weight Container 1 + Waste		_____g
2)	Weight Container 1 + Residue		_____g
3)	Weight of Waste Charged to ZHE ((1) - (2), or weighed directly into vessel)		_____g
4)	Weight of Pre-extraction Collection Device		_____g
5)	Weight of Device and Filtrate		_____g
6)	Weight ((5) - (4))/Volume (mL) of Filtrate	6a_____/_____6b	
7)	_____g of Solid Phase was added to 8)	_____mL of Fluid #1.	
	((3) - (6a))	(20 * (7a))	

Pressure before Tumbling 40 Pressure after Tumbling: 40 Rotation at 30 ± 2 RPM Verified ✓

Time/Date Started Tumbling 15:45 7/22/19 Time/Date Stopped: 10:15 7.23.19 Hours Extracted 18.5 (18 ±2)

Room Temp Start: 23.1 Room Temp. Min. 22.6 Room Temp. Max. 23.4 (23± °C)

Was pre-extracted filtrate recombined with extract _____YES ✓ NO

If "NO" enter volume of filtrate (6) 120

Amount of Fluid (8) _____

Analyst JP/HG Date Filtered: 7.23-19 Time Filtered: 10:33

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-QAMS682 0000072

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

FIGURE 2

ROTARY AGITATION APPARATUS

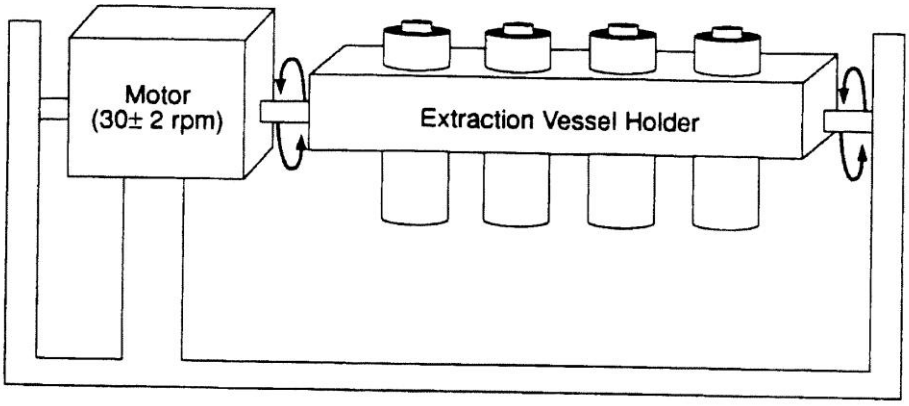

Figure 1. Rotary Agitation Apparatus

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

FIGURE 3

ZHE VESSEL

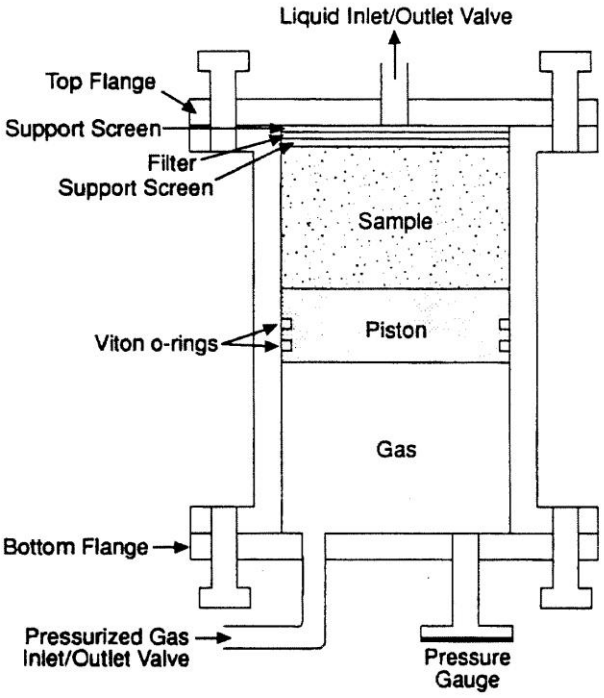

Figure 2. Zero-Headspace Extractor (ZHE)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 020101

Operations Manager: [signature] Date: 1/31/01

QA Officer: [signature] Date: 1.31.01

General Manager: [signature] Date: 2/07/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	EN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with Navy.	EN	09.30.04	09.30.04
03 8270C	Sect. 7.2 Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all references to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address different SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10
09	Sect. 7.4 - Added additional tune information. Sect. 7.6 - Added 100 ml minimum extract vol. & 1 ml IS is added for each 100 ml aliquot. Sect. 7.5.4 - Added RRT information Sect. 9.0 - Added MDL, LOD and LOQ information Table 4 - Added 1,4-Dioxane-d6 Surrogate	LAD	05/11	05/11
10	Sect. 7 - Changed sample volume from 1 ml to 2 ml. Sect. 8 - Added 10% rule for non-DoD clients. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and updated references. Updated Figure 1. Added Addendum 1 - low level 1,4-Dioxane analysis	LAD	05/12	05/12
11	Sect. 1 and 7 - Removed Quickform reporting and added KIMS. Sect. 8 and Table 1 - Added the surrogate 1,4-Dioxane-d8. Throughout - Fixed typos and made minor changes.	LAD	03/13	03/13
12	Sect. 4 - Updated instrument and column models. Sect. 7 - updated calibration levels and prep. Sect. 8 - Added marginal exceedance criteria. Updated ms/MSD acceptance criteria. Tables - Added DoD QSM 5.0 QC Requirements. Updated Fig. 2 & 3	LAD	04/14	04/14
13	Sect. 5 - Added Standards to title. Sect. 7 & Appendix 1 - Updated GC/MS operating conditions. Appendix 1 - Corrected 1,4 dioxane primary and secondary ions. Add 1,4 dioxane-d4 ions. Changed KAS INC to KAS throughout	LAD	03/16	03/16

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 10-Updated method references. Sect 7 -Added percent error calculation and relative std error calculation. Sect 1.1-updated definitions. Sect. 9.0-update. Sect. 7-changed the initial cal. std concentrations. Added compounds to Tables 5 & 7.	LAD	09/17	09/17
15	Sect. 7- updated SSTD30 preparation. Changed CCV to SSTD2.0 and concentration to 2.0 ng/mL. Updated Runlog Example	LAD	01/19	01/19

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process. Refer to section 8 for Method Blank acceptance criteria

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term LOQ must be used for DoD work. Refer to section 9 for specific LOQ/LLOQ verification requirements

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer

to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890.

4.2 Mass Spectrometers (MS): HP5975 or HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: RTX5 SIL MS - 30m, 0.25mm I.D., 25um film thickness, columns (Restek) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

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5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will “boost” the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

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7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument is given a unique identifier)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (Each instrument is given a unique identifier). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (Each instrument is given a unique identifier).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using “Edit Sample Log Table”. For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 2.0, 7.0, 10.0 and 15.0 ng/uL. This is done to determine instrument sensitivity and the

linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds need to be calibrated at higher concentrations. A second standard (Mix B) is prepared containing these compounds. The two standards are combined as in the example below. The full aliquot is used and spiked with the appropriate amount of IS.

Example –

Calibration Mix A is prepared containing ALL analytes at 20 ng/ul.
Calibration Mix B is prepared containing only phenols and phthalates at 20 ng/ul.

For the low standard, 10 ul of mix A and 40 ul of mix B are combined and diluted to 1000 uL with MeCL2. Internal standards are then added prior to analysis.

Calibration Level	Cal-Mix A (All Analytes) Added (uL)	Cal-Mix B (Phenols and Phthalates) Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)	Final Conc. Everything but Phenols and Phthalates (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)
SSTD 0.2	10	40	950	1000	0.20	1.0
SSTD 0.50	25	75	900	1000	0.50	2.0
SSTD 2.0	40	50	310	400	2.0	4.5
SSTD 7.0	70	NA	130	200	7.0	7.0
SSTD 10	100	NA	100	200	10	10
SSTD 15	150	NA	50	200	15	15

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 3.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the six calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

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Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
$\acute{x}$i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD2.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 2.0 ng/uL.

After quantitation of the 2.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 2.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may precede using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap.

This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for

analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.
If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

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8.2 Surrogate Recoveries

The five surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10, Pyrene-d10 and 1,4-Dioxane-d8) must meet the current statistically derived or nominal acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Non-Conformance Report (NCR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

For projects or clients requiring DoD QSM compliance, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained

for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits or nominal limits with the following sporadic exceedance allowances, for DoD clients.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For non-DoD clients corrective action is only taken if greater than 10% of the analytes of interest are outside of the laboratory established acceptance limits.

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8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

All MS/MSD samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8270C and Method 8270D.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. % Error must be ≤ 30%. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation = 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte = 15%; Option 2: linear least squares regression for each analyte: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-15	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

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TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
1,4-Dioxane-d8 (surrogate)
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichlorbenzene
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
1,2,4-trichlorobenzene
1,2,4,5-tetrachlorobenzene
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene
2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

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TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

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TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane-d8	96	66
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
1,3-dichlorobenzene	146	148, 111
1,4-dichlorobenzene	146	148, 111
1,2-dichlorobenzene	146	148, 111
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
1,2,4-trichlorobenzene	180	182, 145
1,2,4,5-tetrachlorobenzene	216	214, 179
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

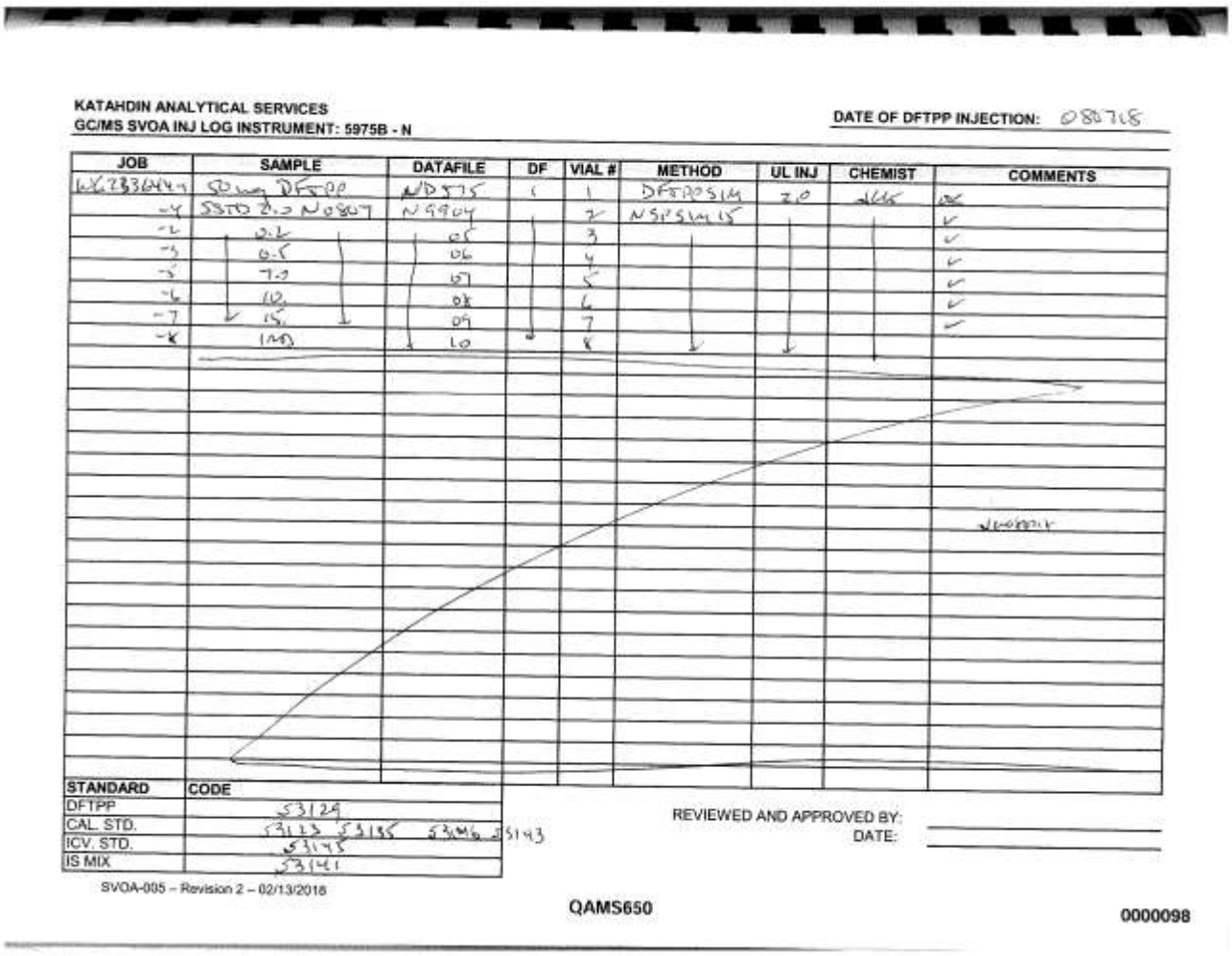
The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5975B - N

DATE OF DFTPP INJECTION: 080718

JOB	SAMPLE	DATAFILE	DF	VIAL #	METHOD	UL INJ	CHEMIST	COMMENTS
LK233644-1	50ng DFTPP	ND575	1	1	DFTPPSIM	2.0	JLG	OK
-4	SSTD 2.0 ND809	N9904		2	NSIPSIM15			✓
-2	0.2	05		3				✓
-3	0.5	06		4				✓
-5	1.0	07		5				✓
-6	10.	08		6				✓
-7	15.	09		7				✓
-8	100	10		8				

STANDARD	CODE
DFTPP	53129
CAL. STD.	53133 53135 53136 53143
ICV. STD.	53145
IS MIX	53141

REVIEWED AND APPROVED BY:
DATE:

SVOA-005 – Revision 2 – 02/13/2018

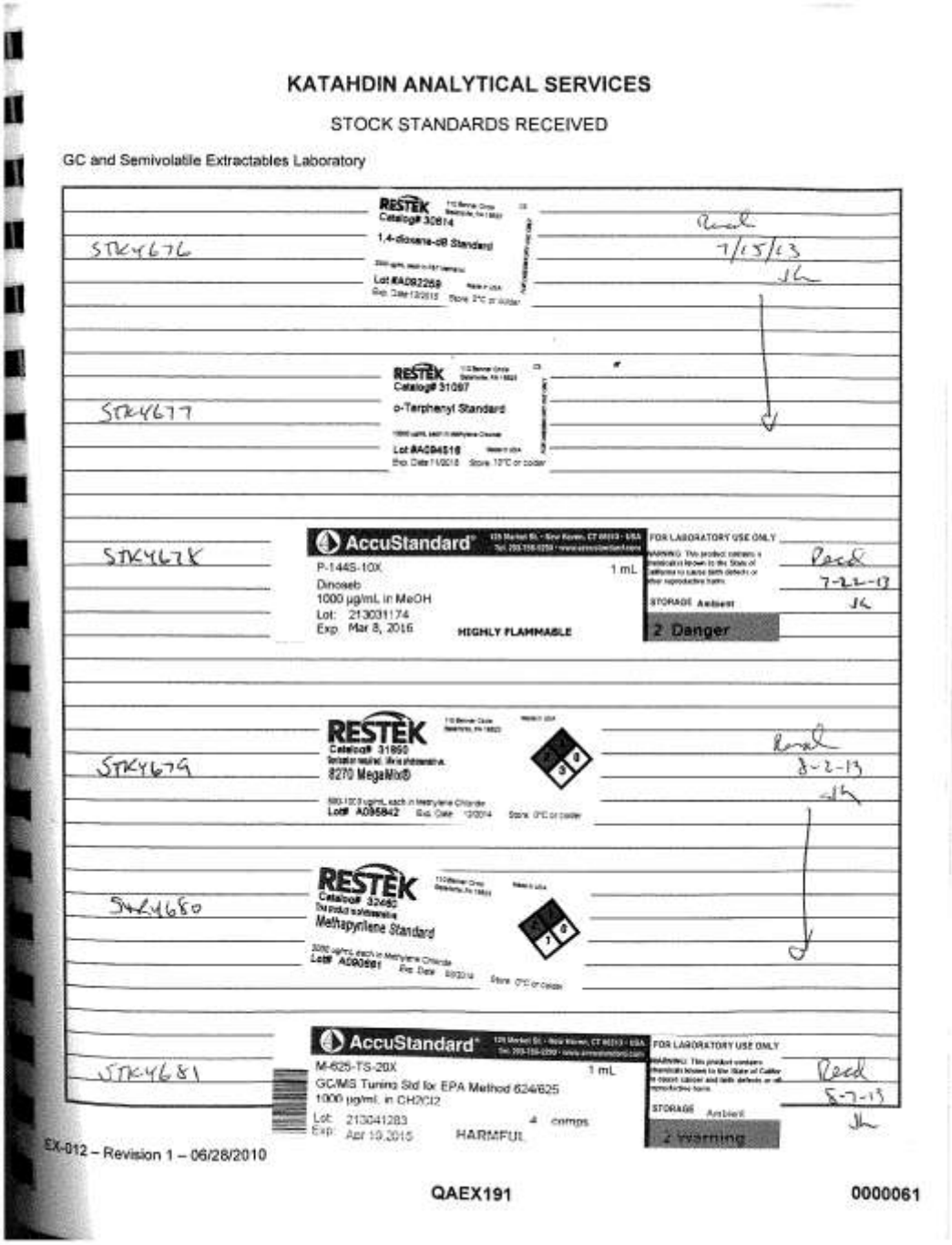
QAMS650

0000098

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GC and Semivolatile Extractables Laboratory

STK4676 — RESTEK Catalog# 30814, 1,4-dioxane-d8 Standard, Lot #AD82259, Exp. Date 12/2015, Store 2°C or colder — Rec'd 7/15/13 JK

STK4677 — RESTEK Catalog# 31097, o-Terphenyl Standard, Lot #AG84518, Exp. Date 1/2016, Store 10°C or colder

STK4678 — AccuStandard, P-144S-10X, 1 mL, Dinoseb, 1000 µg/mL in MeOH, Lot: 213031174, Exp: Mar 8, 2016, HIGHLY FLAMMABLE, FOR LABORATORY USE ONLY, STORAGE Ambient, 2 Danger — Rec'd 7-22-13 JK

STK4679 — RESTEK Catalog# 31850, 8270 MegaMix®, Lot# A095842, Exp. Date 12/2014, Store 0°C or colder — Rec'd 8-2-13 JK

STK4680 — RESTEK Catalog# 32460, Methapyrilene Standard, Lot# A090691, Exp. Date 8/2014, Store 0°C or colder

STK4681 — AccuStandard, M-625-TS-20X, 1 mL, GC/MS Tuning Std for EPA Method 624/625, 1000 µg/mL in CH2Cl2, Lot: 213041283, Exp: Apr 10,2015, 4 comps, HARMFUL, FOR LABORATORY USE ONLY, STORAGE Ambient, 2 Warning — Rec'd 5-7-13 JK

EX-012 – Revision 1 – 06/28/2010

QAEX191

0000061

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

SVOA-004 – Revision 1 – 11/10/10

GC/MS SVOA STANDARD PREP LOGBOOK

Standard ID	Standard Name	Prep Date	Exp Date	Initials	Stock #	Stock Name	Amt Added	Stock Exp Date	Total Vol	Final Conc
S2401	SIM Stock B	4-11-14	6-14-14	JLH	S2367	SIM Pre-Stock B	180	6-14-14	1.8 ml	20 µg/ml
					DK009	MeCl2	1620			
S2402	SIM IND Pre Stock 2	4-15-14	10-15-14	JLH	STD5510	Acid S	~~400~~ 200	4-7-15	1.0 ml	100 µg/ml
					STD5298	B/N	50	↓		
					STD5376	1-Methylnaphthalene	↓	↓		
					STD5495	Benzidine, 3,3'-Dichlorobenzidine	↓	↓		
					STD5314	Composite R3	↓	↓		
					STD5305	1,4-Dioxane	↓	↓		
					STD5145	OLM	20	↓		
					DK009	MeCl2	680			
					STD5315	1,2,4,5-TCBz	100			
S2403	SIM IND Pre Stock 1	4-15-14	10-15-14	JLH	S2402	SIM Prestock 2	100	10-15-14	1.0 ml	10 µg/ml
					DK009	MeCl2	900			
S2404	SIM IND	4-15-14	10-15-14	JLH	S2403	SIM Prestock 1	200	10-15-14	1.0 ml	2 µg/ml
					DK009	MeCl2	800			

Reviewed by/Date:

QAMS521

0000027

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

ADDENDUM 1

LOW LEVEL 1,4-DIOXANE ANALYSIS

The following are differences from the standard 8270 C or D SIM analysis:

GC Operating Conditions – The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP (Sect. 7.4) with the following exceptions:

Column Temperature Program	35°C hold 3 minutes 20°/min. to 300°C
Final Column Temperature hold	300°C
Run Time	16.25 minutes
Scan Start Time	2.3 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

Stock Standards – 1,4-Dioxane and 1,4-Dioxane each at 20 ug/mL

Calibration Standards – Use the above stock standards to prepare calibration standards at concentrations 0.25 ug/mL, 0.50 ug/mL, 1.0 ug/mL, 2.0 ug/mL, 4.0 ug/mL and 6.0 ug/mL. The 1.0 ug/mL is also the continuing calibration verification standard.

Sample analysis – Add 1 uL of internal standard (Section 5.3.2.4) aliquot of sample.

The ions for 1,4-Dioxane are 58 and 88.
The ions for 1,4-Dioxane-d8 are 64 and 96.

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: JC Gomez

Review Date: 3-6-20

SOP Number: SA-213-15

SOP Title: Analysis of SVOA by SIM

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: [signature] **Date:** 3-9-2020

QAO Signature: Jessie Dimond **Date:** 031120

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DN	1.23.01	12301
02 5035	Reorganiced Sections 4, 5, 6, 7 and 8.	HRC	07.02.09	07.02.09
03 5035	Editted Section 6.4.3 to include the addition of 5mL of H2O to Sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06
05	Added 3585 Reference. Sections 6.1.2.3, 6.4.3 and 7.2.2: changed 20mL to 5mL.	LAD	09108	09/08

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Small changes to sections 3.2, 4.2.2.1, and 7.1.1 to address the differences between Purge and Trap Autosamplers. Added references.	LAD	03/12	03/12
07	Sec 6.1.1.2 - Changed 20mL to 5mL. Secs 6.1, 6.2, 6.4: edited and added steps for clarity. Changed required balance accuracy from 0.1 g to 0.01g throughout. Minor formatting and grammatical changes. Updated references	LAD	03/18	03/18
08	Changed 200 ug/kg to 150 ug/kg. Changed some DI H2O to read DI H2O. Sect. 4.2.1 - updated purge instrument requirements. Other minor edits.	LAD	01/20	01/20

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-08**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-08**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 150 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent by method 3585. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 5030, 5035 and 3585. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 150 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 150 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 150 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared in n-hexandecane according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free laboratory

reagent grade water or water miscible solvent and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Purge and Trap autodampler systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the

sample. The device should also be capable of introducing at least 5 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240°C - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 29 - 30 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.3 Micro syringes - 10-, 100-µL.

4.3.4 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.1 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS AND STANDARDS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low concentration soil samples

Sodium bisulfate preservation is used in the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

Water and subsequent freezing preparation of vials is used in the collection of low concentration soil samples known to contain carbonate minerals which may effervesce upon contact with an acidic preservation solution and which are to be analyzed by the closed-system purge-and-trap equipment described in Method 5035. This type of preservation is typically done in the lab after Encore samplers are received from the field. This must be done within 48 hours of sampling.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 The preservative is added to each vial prior to shipping the vial to the field. Add 5 mL of 20% sodium bisulfate solution or 5 mL of water to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is

negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.1 g and record it in the logbook.

6.1.2 High concentration soil samples in methanol:

6.1.2.1 When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40-mL glass vials with septum seals (see Sec. 4.4).

6.1.2.2 The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.2.3 Add 5 mL of methanol to each vial.

6.1.2.4 Seal the vial with the screw-cap and septum seal.

6.1.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.2.6 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.2.2, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.1.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™ , and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 5 mL of water and storage at -10° (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples.

7.0 PROCEDURES

This section describes procedures for the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.1 Low concentration soil method (Approximate concentration range of 5 to 150 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Purge and Trap Autosampler Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

7.1.1.1 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.2 Carry out the purge-and-trap procedure as outlined in Secs. 7.1.2. to 7.1.4.

7.1.2 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain sealed in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial

in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water, the internal standards, and the surrogate compounds. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

7.1.3.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 1 to 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration method for soil samples with concentrations generally greater than 150 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 5.0 g (wet weight) of sample to a tared 40-mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.1 g. Add 5 mL of methanol to the vial containing the sample and shake for two minutes.

NOTE: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.3 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.4 For each new lot of methanol, add an appropriate aliquot of the methanol to 40 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared

using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane and shake for two minutes.

7.3.2 For oily samples that are soluble in methanol if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), V (2015) and VI, Method 5035

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), V (2015) and VI, Method 5035A

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 3585, SW-846, USEPA, Revision IIIB, Nov. 2004.
Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

LIST OF TABLES AND FIGURES

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-08	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D

Prepared By: Semivolatile Group Date: 02.11.09

Approved By:

Department Manager: [signature] Date: 2-11-09

Operations Manager: Deborah J. Radeau Date: 2.11.09

QA Officer: Susie Dimond Date: 02.10.09

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated to reflect DOD QSM version 4.1 compliance and new standard preparation procedures.	EN	08/09	08/09
02	Edited Sections 5.3.2 and 7.5.1 to reflect current calibration levels. Added Table 2 - DoDQSM QC criteria. Renumbered Tables 2-6. Added references.	LAN	08/10	08/10
03	Added new instrumentation. Added LOD and LOQ verification information. Added DDT breakdown equation. Added TNI reference. Minor changes to reflect current techniques.	LAN	01/12	01/12
04	Sect. 1 & 7 - Changed reporting through quickform to reporting through KIMS. Sect. 8 - Added Marginal Exceedance criteria.	LAN	04/13	04/13
05	Sect. 7 - Corrected typo and formatting. Sect. 10 - Added and updated references. Added Table 3 - DoDQSM5.0 QC Requirements Renumbered Tables 3→8.	LAN	07/14	07/14

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Table 1- Corrected ICV acceptance criteria. -Changed KAS INC. to KAS throughout.	LAD	08/15	08/15
07	Sect. 5 - Added standards to title. Sect. 7 - Updated GC/MS operating parameters added resolution criteria for structural isomers.	LAD	03/16	03/16
08	updated for SW846 Update V references.	LAD	10/16	10/16
09	Sect. 7 - Added % Error calculation Sect. 9 - Added LLOQ reference	LAD	03/17	03/17
10	Sect. 1, 9 and Table 1 - Added LLOQ definition and verification acceptance criteria, clarified PQL, LOQ and LLOQ. Added SC does not allow for non-linear calibration model.	LAD	06/17	06/17

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-226-10, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-226-10, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270D.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions:

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation Of Aqueous Samples For Extractable Semivolatile Analysis", SOP CA-512, "Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890, 6890 and/or 6890N

4.2 Mass Spectrometers (MS): HP5973, HP5972, HP5970 and/or 5975B

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As and HP 7683s

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W

Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

4.10 1.8 mL vials with 350uL inserts

4.11 Crimp tops with Teflon lined septa

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared on an as needed basis (but not less than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should <u>not</u> be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 5 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source

other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 5 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument has a unique ID)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID. This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID.

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:
    - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
    - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the “Position and run” command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30 to 60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	base peak, 100 % relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

The following are the GC/MS operating conditions for injection of DFTPP.

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0-6.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using “Edit Sample Log Table”. For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

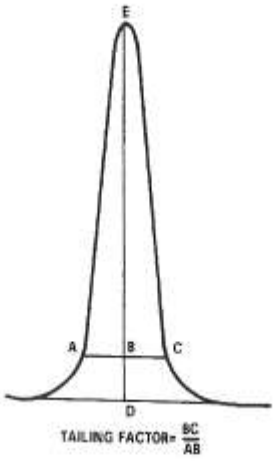
If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

Tailing Factor = $\frac{BC}{AB}$

.where: AC = the width at 10% height
DE = height of the peak
B = the height at 10% of DE

Example:



Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

The Enviroquant system uses the following formulas to determine the % breakdown of DDT and Endrin -

% Breakdown DDT = $\frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} * 100$

% Breakdown Endrin = sum of degradation peak areas (aldehyde + ketone) * 100
sum of all peak areas (endrin + aldehyde + ketone)

Degradation of DDT to DDE and DDD should not exceed 20%.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270D

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.

After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the Mean RRF and %RSD for all analytes. If information is needed concerning the use of these programs, consult the Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for

each target analyte must be less than or equal to 20%.

It is recommended that a minimum response factor (Table 8) for target analytes be achieved as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Therefore the minimum response factors in Table 8 must be verified at the lowest calibration level.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 20% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 20%, then a calibration option outlined in section 11 of method 8000D will need to be employed.

Option 1 (Section 11.5.2 of method 8000D - Rev. 4, 07/14), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2. This is calculated by either calculating r or squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control".

Corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

Option 2 (Section 11.5.3 of method 8000D - Rev. 4, 07/14), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration models for compliance work originating in their state.

If more than 10% of the compounds in the initial calibration exceed the 20% RSD limits and do not meet the minimum correlation coefficient of determination criteria in option 1 or 2, the GCMS system is considered out of control and the calibration must be repeated. Note: Maintenance may have to be performed.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective action, including ion source cleaning or repair and column removal or replacement.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate

the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.5.2.3 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. The percent difference for each target analyte must be less than or equal to 30%. For clients requiring DOD criteria, all project analytes must be within +/- 20% of

true value.

7.5.2.4 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.

After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences.

- All target analytes must have a % difference of +/- 20%D in order to be considered in criteria.

- All target analytes should meet the minimum RRF criterion as in ICAL (Table 8) in order to be considered in criteria.

These conditions must be met before method blank and/or sample analysis can begin.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.

- Change the septum; clean the injection port; install a clean, silanized

quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different gas chromatographic retention times. Sufficient gas chromatographic resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis
by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add a minimum of 100 uL of sample extract and 1.0 uL (for each 100 ul of sample) of the 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who analyzed the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Tables 1, 2 and 3). These tables give acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual

integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.
For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.

7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.

7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.

7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.

7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

7.8.6 Take out the liner and rinse it thoroughly with toluene.

7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.

7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform

other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Tables 1, 2 and 3 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Tables 1, 2 and 3, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Tables 1, 2 and 3 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Tables 1, 2 and 3 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.
Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14
- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. For DoD ELAP work, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the mid-point of the initial

calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. South Carolina does not allow for marginal exceedances for compliance work originating in their state.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also

can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.
Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Tables 1, 2 and 3 for a summary of QC requirements, acceptance criteria, and corrective actions. Tables 1, 2 and 3 criteria are intended to be guidelines for analysts. The table does not over all possible situations. If any of the QC

requirements are outside the recovery ranges listed in Tables 1, 2 and 3, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Tables 1, 2 and 3 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8270D. Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IV and V, July 2014, Method 8270D.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IV and V, July 2014, Method 8000D

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD 20% for all compounds. If not met: Option 1) Linear least squares regression: r ≥ 0.995 Option 2) Non-linear regression: coefficient of determination (COD) r^2 ≥ 0.99 (6 points for second order) Up to 10% target analytes may be outside of the above criteria % Error ≤ 30% Refer to section 7.5.2.1 for additional information.	Perform instrument maintenance if necessary. Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	All target analytes: ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL/LLOQ	1) Investigate source of contamination 2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. 2) If an LCS/LCSD was performed and only one was unacceptable, narrate. 3) If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. 4) If the LCS rec. is high but the sample results are <PQL, narrate. 5) Otherwise, reprep a blank and the remaining samples.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Current statistical limits	1) Check chromatogram for interference; if found, flag data 2) If not found, check instrument performance; if problem is found, correct and reanalyze 3) If still out reextract and analyze sample 4) (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. 2) (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation = 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte = 15%; Option 2: linear least squares regression for each analyte: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3

DOD QSM 5.0/5.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 4

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-226-10	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine (not on TCL list)
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)
Dinoseb (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 6

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

RSD 20% for all compounds.
If not met:
Option 1) Linear least squares regression: $r \geq 0.995$
Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order)
Up to 10% of target analytes may be outside of the above criteria
Refer to section 7.5.2.1 for additional information.
Recommended minimum RF criteria for analytes listed in Table 8.

Continuing Calibration Check Criteria

All target analytes: $\leq$ 20%D
Recommended minimum RF criteria for analytes listed in Table 8.

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 7

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	107,108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	107,108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 7 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120

TABLE 7 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Dinoseb	211	163, 240
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 7 after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

Table 8

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100

Table 8

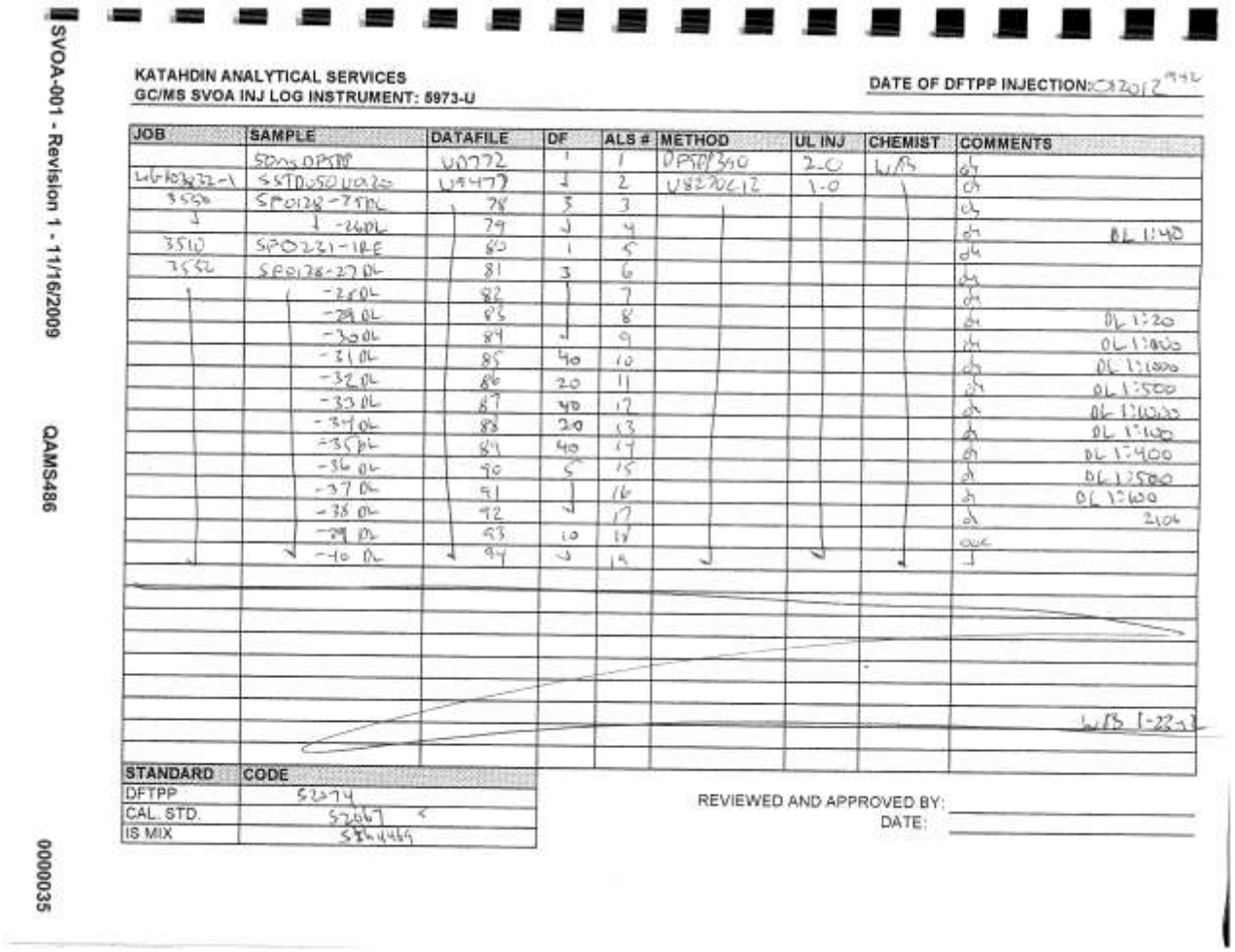
RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION (CONT.)

Semivolatile Compounds	Minimum Response Factor (RF)
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



SVOA-001 - Revision 1 - 11/16/2009

QAMS486

0000035

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION: 3/20/12

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	SDG DFTPP	U0772	1	1	DFTPP350	2.0	WB	
L1603872-1	SSTD050 U0773	U0777	1	2	U8270C-12	1.0		
3550	SP0128-7 5DL	78	5	3				
1	1 -2 5DL	79	1	4				RL 1:40
3510	SP0231-1RE	80	1	5				
3552	SP0128-2 7 DL	81	3	6				
	-2 5DL	82		7				
	-28 DL	83		8				DL 1:20
	-30 DL	84		9				DL 1:100
	-31 DL	85	40	10				DL 1:1000
	-32 DL	86	20	11				DL 1:500
	-33 DL	87	40	12				DL 1:1000
	-34 DL	88	20	13				DL 1:100
	-35 DL	89	40	14				DL 1:400
	-36 DL	90	5	15				DL 1:500
	-37 DL	91	1	16				DL 1:600
	-38 DL	92	1	17				2.04
	-39 DL	93	10	18				DUP
	-40 DL	94	1	19				
								WB 1-23-12

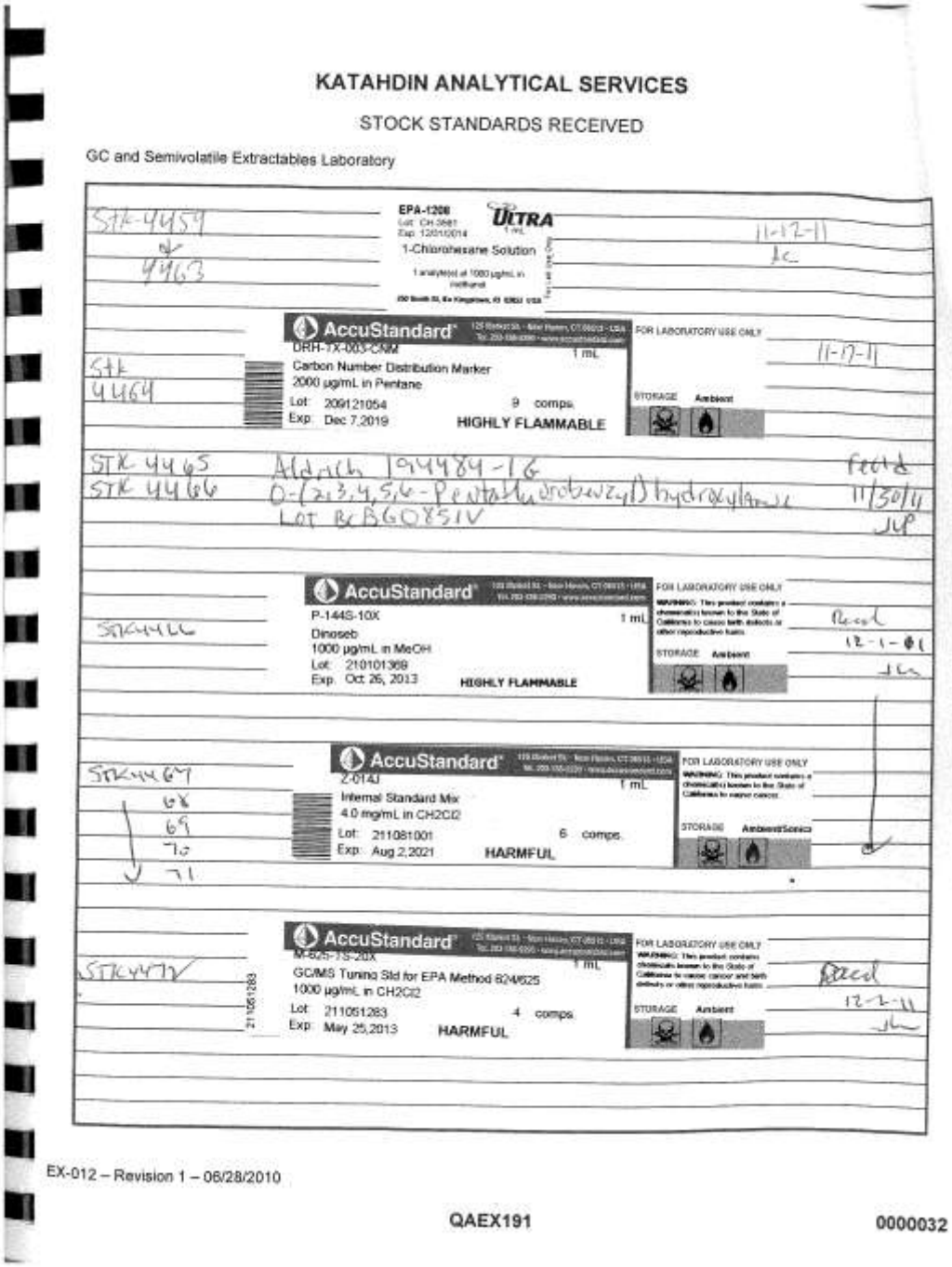
STANDARD	CODE
DFTPP	52674
CAL. STD.	52667
IS MIX	52669

REVIEWED AND APPROVED BY: _____
DATE: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



EX-012 – Revision 1 – 06/28/2010

QAEX191

0000032

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

SVOA-004 – Revision 1 – 11/10/10

GC/MS SVOA STANDARD PREP LOGBOOK

Standard #	Standard Name	Prep Date	Exp Date	Initials	Stock #	Stock Name	Amt Added	Stock Exp Date	Total Vol	Final Conc
S2072	Pyrene-d10	1-12-12	7-12-12	JLK	S2069	Pyrene-d10 Stock	353	1-12-13	4 mL	200 µg/mL
					DF335	MeCl2	To 4 mL			
S2073	2,4-DAP	1-12-12	7-12-12	JL	S2070	2,4-DAP Stock	214	1-12-13	4 mL	200 µg/mL
					DF335	MeCl2	To 4 mL			
S2074	DFTPP Soln	1-13-12	7-13-12	JL	STK4474	M-GK-B-2xx	250 ~~12-12-12~~	12-12-12	10 mL	25 µg/mL
					STK4354	Benzidine	125	7-14-12		
					DF335	MeCl2	To 10 mL			
S2075	SIM Stock A w/(1,2,4,5-TCB)	1-17-12	5-31-12	JL	STK4453	8270 mega mix	108	12-20-12	5.4 mL	20 µg/mL
					S1997	OLM Compds	↓	8-12-12		
					STK4255	1,2,4,5-TCB	↓	1-17-13		
					STK4357	1,4-Dioxane	54	7-17-12		
					STK4458	3,3-Dichlorobenz	↓	11-23-12		
					STK4384	1,4-Dioxane-d8	540	5-31-12		
					STD4222	2-Methylnaph-d10	540	8-12-12		
					S2071	Fluorene-d10	↓	7-13-12		
					S2072	Pyrene-d10	↓	↓		
					S2073	2,4-DBP	↓			
					DF335	MeCl2	To 5.4 mL			
S2076	SIM IND	1-17-12	6-2-12	JL	SV2517	8270 Spike	72	6-2-12	1.8 mL	2/4 µg/mL
							1728			

Reviewed by/Date:

QAM5452

0000051

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: J.C. Gomez

Review Date: 1-22-19

SOP Number: CA-226-10

SOP Title: Analysis of Semivolatile Organic Compounds by Capillary column GC/MS: SW 846 Method 8270D

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-24-19

QAO Signature: Leslie Dimond **Date:** 01.25.19

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: JC Gomez

Review Date: 3-6-20

SOP Number: CA-226-10

SOP Title: Analysis of SVOA by Cap Col : 8270D

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: [signature] **Date:** 3-9-2020

QAO Signature: Leslie Dimond **Date:** 03/11/20

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Prepared By: Peter Lemay Date: 7/96

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Dennis F. Hufnagle Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
CR 8081A	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1.	DN	1.22.01	1/22/01
03 8081A	Changes to comply with South Carolina requirements – added linear calibration option, retention time window criteria & other minor changes to surrogate criteria.	DN	5.21.01	5.21.01
04 8081A	Changed to practice of reporting higher value. Other minor changes to Table 1 & 2, section 7.5.3 and section 7.4.3.	DN	5.21.02	5.21.02
05 8081A	Added definitions and information for the new data processing system. Replaced several figures with updated ones.	MRC	05.04.04	05.04.04
06 8081A	added alternative CV Conc. changed data checklist minor changes throughout added wording to section 8	LAD	3 03/05 LAD	3 03/05 6-13-06

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added retention time window criteria. Sect. 7.4.2 - added to Shake samples before vialing.	LAD	03/06	03/06
08	Sect. 1.4 - Updated to include waste streams. Sect. 4.5 - removed balance. Changed makeup gas from N_2 to Ar Me. Changed TC from 5 to 2 peaks. Updated column confirmation. Changed corr. coef. to coeff of determination	LAD	06/07	06/07
09	Added extraction method 3535 for AQ Samples. Updated method references. Added Katahdin Analytical Environmental Health and Safety Manual. Changed number of peaks quantitated for Toxaphene to 3 & 5. Added S.C. clarification on marginal exceedences.	LAD	02/09	02/09
10	Changes made to Sections 4.1, 7.4, 7.5, 8.0 and 10.0 for compliance with DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM version 4.1 QC Requirements. Updated Figure 1 and 2.	LAD	04/10	04/10
12	Edited Sections 5.25, 7.2.1, 7.2.2, 7.2.3, 8.2, Table 1, Table 2 and Figure 3 to include calibration PQL and Spike information for toxaphene and technical Chlordane. Updated Data Review checklist.	LAD	06/11	06/11
13	Added extraction method 3546. Removed Quickform reference. Added reporting from KIMS. Added marginal exceedence information. Updated references. Updated Fig.s 1 and 2.	LAD	02/13	02/13
14	Sect. 2 - Added GPC cleanup. Sect. 7 and Table 1 - Added average calibration model. Sect. 8, Table 1 and Table 2 - Removed nominal Limits except for TC. Soil. Added Table 3 - DoDQSM 5.0 QC Requirements. Updated Figure 2 - Review Checklist	LAD	06/14	06/14

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
15	Sect. 5 & 7 – Changed concentration of Endrin in EVAL standard from 0.2 to 0.1 ug/ml. Sect. 7- Added South Carolina requirement of not allowing non-linear calibration models. Figure 2 – Updated Review Checklist	LAD	07/15	07/15
16	Sect. 5 - Added Standards to title. Sect. 7- changed T.C. calibration from 3-5 peaks to 2-5 peaks. Added ICV for TDx and T.C. Table 1- corrected Calib. Criteria. Corrected typos	LAD	03/16	03/16
17	Sect. 7 - Added %Error calculation Sect. 9 - Added LLOQ reference	LAD	03/17	03/17
18	Sect. 1, 8 and Table 1 - Added LLOQ definition and acceptance criteria. Clarified PQL, LOQ and LLOQ. Sect. 7 - updated CCV Criteria. Updated Data Review Checklist	LAD	06/17	06/17
19	Sect. 1 and Table 3 - Added additional compounds. Removed Table 2 - DoDQSM 4.2 QC Requirements. Updated References. Updated Logbook Page Example	LAD	07/18	07/18
20	Minor changes to Sect. 7	LAD	10/19	10/19

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for Pesticides by EPA Method 8081B, as performed by Katahdin Analytical Services including sample analysis by Gas Chromatography-Electron Capture Detector (GC-ECD), data review, standard preparation and instrument calibration.

It is applicable to the following compounds:

Aldrin	Sulfate
Alpha-BHC	Endrin
Beta-BHC	Endrin aldehyde
Gamma-BHC	Heptachlor
Delta-BHC	Heptachlor epoxide
Chlordane	Toxaphene
4,4'-DDT	Endrin ketone
4,4-DDE	Methoxychlor
4,4'-DDD	Technical chlordane
Dieldrin	trans-Nonaclor
Endosulfan I	Mirex
Endosulfan II	

Though not listed in the method, the following compounds can be analyzed for by this method:

cis-Nonachlor	2,4'-DDT
2,4'-DDD	Hexachlorobenzene
2,4-DDE	Oxychlordane

1.1 Definitions

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

INDEPENDENT CALIBRATION VERIFICATION (ICV): A verification of the ratio of instrument response to analyte amount. ICV solutions are prepared from stock solutions which are independent from the stock solutions used to prepare the calibration standards.

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however, a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory can make accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

HP ENVIROQUANT: A data acquisition system that is used to collect chromatographic data.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pesticides by method 8081, current revision. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8081, current revision, to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into

the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

2.0 SUMMARY OF METHOD

2.1 Method 8081 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8081 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8081 may also be performed on samples that have undergone cleanup. Method 3640A, Gel Permeation Chromatography (GPC), is used to eliminate interferences in the analysis of most soil extracts. Method 3660, Sulfur Cleanup may also be used to cleanup extracts prior to analysis.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a problem in pesticide determinations when using the electron capture detector. Common flexible plastics contain various amounts

of phthalates. Care must be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 6890 or 5890 series I or II or 6890 connected to the Turbochrom or Enviroquant data system, or equivalent.

4.1.2 Columns: Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron Capture Detectors (ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS AND STANDARDS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in a separate logbook.

5.2.3 Pesticide working standards: Prepared by diluting the stock mix of 2000 ug/ml that contains all single component pesticides into hexane to give final concentrations of 0.005, 0.01, 0.025, 0.05, 0.10, and 0.25 ug/ml. The mix, referred to as INDAB, also contains two surrogates: Tetrachloro-m-xylene and Decachlorobiphenyl, which are at the same concentrations as the pesticides.

5.2.4 Independent Calibration Verification Standard: Prepared as above using a standard independent of the calibration standards.

5.2.5 Multicomponent Pesticide Working standards: Toxaphene is prepared by diluting the Toxaphene stock solution into hexane to give final concentrations of 0.10, 0.25, 0.50, 1.0, 2.5 and 10 ug/ml. Technical chlordane is prepared similarly except to a concentration of 0.05, 0.10, 0.25, 0.50, 1.0, and 2.5.ug/ml.

5.2.6 Evaluation Mix: Prepared by diluting the stock solution to a concentration of 0.20 ug/mL for 4”4”-DDT and 0.1 ug/ml for Endrin.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURES

EXTRACTION - Refer to the appropriate SOP for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3545, 3546 or 3550.

7.1 INSTRUMENT CONDITIONS

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:	Makeup flow:	60 ml/min Nitrogen or Ar/Methane
	Column flow:	3.75 ml/min
	Injector Temp:	200
	Detector Temp:	300
	Oven Ramp:	170(0) - 5/min - 300(10)
	Run time:	24 min
	Injection size:	2 uL

7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. At least a six-point calibration standard mix of the INDAB mix listed in Reagents Section 5.2.2 is prepared along with a single point standard of Toxaphene and Technical Chlordane.

7.2.2 A multicomponent pesticide (toxaphene and technical chlordane) calibration must be analyzed when a multicomponent LCS is required for DoD QSM or if there are detects in samples.

7.2.2.1 Toxaphene is calibrated using the 5 to 10 major peaks of the standard. The Target system will calculate a peak height for each 5 to 10 peaks. A calibration curve is prepared in Target using the peak heights of the 5 to 10 peaks against the concentration of the standard.

7.2.2.2 Technical Chlordane is calibrated using 2 to 5 major peaks of the standard. The Target system will calculate a peak height for each peak. A separate calibration curve for each of the 2 to 5 peaks is prepared in Target using the peak height against the concentration of the standard.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard.

7.2.3 Linear calibration using the average calibration factor – requires at least 5 calibration levels.

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.2.4 Linear calibration using a least squares regression - requires at least 5 calibration levels.

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995.

7.2.5 A non-linear calibration using a second order polynomial (quadratic fit) equation - requires at least 6 calibration levels.

The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = instrument response
b = slope of the line
x = concentration of the calibration standard
c = the intercept

In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990.

The ICAL must be successful before any samples or other QC check samples can be analyzed.

Note: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.2.6 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|}/(\text{n-p})$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.2.7 The INDAB mix, toxaphene and technical chlordane calibration curves must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.2.8 The working calibration curve must be verified on each 12-hour shift that samples are to be analyzed by injecting the mid-point calibration standard.

7.3 RETENTION TIME WINDOWS

7.3.1 Three injections of all single component standard mixtures and multiresponsive products throughout the course of a 72-hour period.

7.3.2 The standard deviation of the three retention times is calculated for each single component standard. For multiresponsive products, a major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. For multiresponsive analytes, the analyst should use the retention time window for each of the individual peaks but also rely on pattern recognition.

7.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: $\pm$ 0.05 for Heptachlor, Aldrin and all BHC compounds, $\pm$ 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of $\pm$ 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.4 GAS CHROMATOGRAPHIC ANALYSIS

7.4.1 Before calibration is performed, and at the beginning of each 12 hour shift, the system is evaluated for analyte degradation by the analysis of a standard mix containing only endrin and 4,4'-DDT, often called an evaluation mix (EVAL):

COMPOUND	CONCENTRATION
Endrin	0.10 ng/uL
DDT	0.20 ng/uL

The % breakdown of DDT and the % breakdown of Endrin is calculated using the following formulas (PH = Peak Height):

$$\textbf{\%}\ \text{Breakdown DDT} = \frac{(PH\ [DDD] + PH\ [DDE])}{(PH\ [DDD] + PH\ [DDE] + PH\ [DDT])} * 100$$

$$\textbf{\%}\ \text{Breakdown Endrin} = \frac{(PH\ [\text{Endrin Aldehyde}] + PH\ [\text{Endrin Ketone}])}{(PH\ \text{of}\ [\text{Endrin Aldehyde}] + PH\ \text{of}\ [\text{Endrin Ketone}] + PH\ \text{of}\ [\text{Endrin}])} * 100$$

The breakdown of either DDT or Endrin in the evaluation mix cannot exceed 15%. If there is breakdown of either compound exceeding 15% before starting a calibration, instrument maintenance must be performed. A calibration can not be run until the evaluation mix meets the acceptance criteria. If the exceeding breakdown occurs during the analysis sequence, then any samples analyzed after a failing evaluation mix must be reanalyzed. Reanalysis can not resume until after an acceptable evaluation mix.

7.4.2 Gently shake sample extracts before vialing for analysis.

7.4.3 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 uL injection volumes.

7.4.4 Samples are analyzed in a set referred to as an analysis sequence.

Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 0.05ppm concentration calibration verification standard (CV).

The CCV calculated concentration must not exceed ± 20% Difference or Drift from the true value.

Each sample analysis must be bracketed with an acceptable initial calibration and closing CV or an opening CV and a closing CV for each 12-hour shift.

The closing CV standard concentration is 0.025ppm.

The CV standard must be injected at intervals of not less than once every ten field samples and at the end of the analysis sequence.

If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard

analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >20%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present.

In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.4.5 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.4.6 The identification of Pesticides is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the absolute retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.4.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a florisil cleanup (method 3620) and/or a sulfur cleanup (method 3660). Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.4.9 When a GC system is determined to be out of control because either a CV can not pass or a six point calibration does not meet the coefficient of determination criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, replacing the Y connector, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.

7.4.10 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibrated method.

7.4.11 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.4.11.1 Water: Concentration (ug/L) = (C) (Vt)/(Vs)

7.4.11.2 Soil / Sediment:Concentration (mg/kg) = (C) (Vt)/(Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.5 Data Review

7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.7.

7.5.2 Surrogate recovery

All recoveries must meet the most recent laboratory established acceptance limits, which are listed on the Laboratory Surrogate Acceptance Limit sheet.

For DoD work, the surrogates must meet the acceptance limits in the DoD QSM.

The sample is evaluated for recoveries of the two surrogates. The recoveries of both surrogates are evaluated on both the primary and secondary column. The higher recovery from both columns is reported on the analytical report for both surrogates. The sample chromatogram is reviewed for any interferences before determining whether to accept a sample based on the surrogate recoveries. If the surrogate recovery is affected by matrix interference, the sample result may be accepted with narration. If the recovery of one surrogate is outside of the laboratory established acceptance limit on one or both columns, and the second is acceptable, the data is narrated. If the recoveries for both surrogates are not acceptable because the recoveries are high and the sample does not contain any analytes above the PQL, the data is narrated. If the recoveries for both surrogates are low and there is no apparent matrix effect, the sample is reextracted.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD work, both surrogates must pass the acceptance criteria.

7.5.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.4.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.5.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

All flagged data must be discussed in the narrative

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV.

The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

7.6 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on

the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.1.1 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected

above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.1.2 LCS, MS/MSD and Surrogate Spike concentrations:

The LCS and the MS/MSD are spiked with the twenty single component pesticides at the same concentration. Toxaphene and technical chlordane LCSs are prepared when required by the project. The spike concentrations are:

	WATER ug/L	SOILS ug/Kg
Individual Pesticides	0.50	16.7
Toxaphene	10	330
Technical Chlordane	10	330

The surrogate spike concentrations in the final extract are:

	WATER ug/L	SOILS ug/Kg
Tetrachloro-m-xylene(TCX)	1.0	33.3
DCB	1.0	33.3

8.1.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For DoD work, the recoveries may be compared to DoD QSM acceptance limits. The nominal limits 50-150 are used for technical chlordane in soil matrix.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. **South Carolina does not allow for marginal exceedances for compliance work originating in their state.**

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They

also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

DoD work requires Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.1.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.2 Non-conformance Report (NCR): Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report must be initiated as soon as possible.

8.3 Contingency for handling out-of-control or unacceptable data – Contact Department Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by

the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion. For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8081 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 8081B, Revision 2, February 2007, Final Update IV to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846.

Method 8000D, Revision 5, March 2018, Final Update V to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January 2017

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

LIST OF FIGURES

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL / LLOQ	(1) Investigate source of contamination (2) Evaluate the samples and associated QC:i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Statistically derived limits. Nominal limits 50-150 for technical chlordane (soil). Note that limits wider than 70-130% are not allowable for some states, programs or clients, i.e. South Carolina. See also section 8.4 of this SOP for more information on allowable exceedances	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	If calibration curve previously analyzed, analyze daily before samples and after every 10 samples.	± 20% D	(1) Evaluate the samples: If the %D>+20% and sample results are <PQL, narrate. If %D>±20% only on one channel, narrate. If %D>±20% for the closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One per sample delivery group (SDG) or every 20 samples.	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
6 pt of INDAB mix with mid-pt cal of Toxaphene and Chlordane	Initial cal prior to sample analysis	5 or 6pt calibration Linear (r) ≥ 0.995 or %RSD ≤ 20% Non-linear (r^2) ≥ 0.990 % Error ≤ 30% or RSE ≤ 30% (≤30% for poor performers)	(1) Repeat Initial calibration (2) If single pt cal Toxaphene, or Chlordane is identified in analysis of sample, 6 pt calibration run of identified compound with reanalysis of sample.
Independent calibration verification	Once after Initial calibration	± 20 % D	Reanalyze standard Reprep standard Reprep standard from fresh stock.
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	One time per analyst initially and annually thereafter.	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDTand Endrin is each = 15%.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-20	METHOD 8081, current revision
Apparatus/ Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
QC - Continuing Calibration	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

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FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services

GC Laboratory Instrument Runlog
Instrument: GC01
Amount Injected: 2 uL
Column Numbers: 473/474
Method: SW846 8011 / 8081 / 8082 / 8151
(circle) EPA 504.1 / 556 / 608

Standard	Standard ID
EVAL	ID 9029
IWDA3 0.05	ID 9158
TOX 1.0	ID 9025
TC 0.5	ID 9114
AR1660 1.0	ID 9170

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
7-16-18	AC	LLG10 300	3550 Tissue MDL6	Y	NA	Pest217	
		301	7	Y			
		302	8	Y			
		303	9	Y			
		304	LCS	Y			
		305	8extra 0.025	Y			
		306	Hexane	N			Tissue Matrix
		307	↓	Y			↓
7-18-18		308	Prime	N	W6232352	CPCTA24	
		309	CV50	Y			
		310	W6232352-1	Y			
		311	-2	Y			
		312	SL6390-4	Y			
		313	-10	Y			
		314	CV25	Y			
7-19-18			Change Liner				
		315	Prime	N	W6232427	Pest217	
		316	TOX 1.0	Y			
		317	TC 0.5	Y			
		318	AR1660 1.0	Y			
		319	EVAL	Y			
		320	IWDA3 0.05	Y			
		321	W6232329-1	Y			608 QC
		322	-2	Y			↓
		323	SL6480-9	Y			
		324	-12	Y			
		325	3510 LOD1				
		326	LOD2				
		327	LOQ				
		328	LOQ2				

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FIGURE 2

DATA REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Int.* _____) **DueDate** _____ .

Client:	**Verbal Review**	**Primary Review**	**Secondary Reveiw**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT* ***ND's*** *to -* ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitation Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐) .	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD O LAB O	☐ FORM 6 (s) DOD O LAB O
☐ FORM 4 (s)	☐ FORM 7 (s) DOD O LAB O
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ O MDL O LOD O
☐ FORM 8 (s)	☐ Flagging B O L O M O C O
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ O MDL O LOD O	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD O LAB O	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD O LAB O	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	**Exceedences**
< 11	**0**
11 to 30	**1**
31 to 50	**2**
51 to 70	**3**
71 to 90	**4**
> 90	**5**

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 3

PQLS FOR METHOD 8081

Parameter/Method	Analyte	Practical Quantitation Level (PQL)	
		Waters (ug/L)	Soils (ug/kg)
Organochlorine	Aldrin	0.05	1.7
Pesticides	Alpha BHC	0.05	1.7
	Beta BHC	0.05	1.7
SW3510 / SW8081B (W)	Delta BHC	0.05	1.7
SW3520 / SW8081B (W)	Gamma BHC (Lindane)	0.05	1.7
SW3540 / SW8081B (S)	Chlordane	0.50	17
SW3545 / SW8081B (S)	alpha-Chlordane	0.05	1.7
SW3546 / SW8081B (S)	gamma-Chlordane	0.05	1.7
SW3550 / SW8081B (S)	4,4'-DDD	0.10	3.3
	4,4'-DDE	0.10	3.3
	4,4'-DDT	0.10	3.3
	Dieldrin	0.10	3.3
	Endosulfan I	0.05	1.7
	Endosulfan II	0.10	3.3
	Endosulfan Sulfate	0.10	3.3
	Endrin	0.10	3.3
	Endrin Aldehyde	0.10	3.3
	Endrin Ketone	0.10	3.3
	Heptachlor	0.05	1.7
	Heptachlor Epoxide	0.05	1.7
	Methoxychlor	0.50	17
	Toxaphene	1.00	33
	Technical Chlordane	0.50	17
	trans-Nonaclor	0.10	3.3
	Mirex	0.10	3.3
	cis-Nonachlor	0.10	3.3
	2,4'-DDD	0.10	3.3
	2,4-DDE	0.10	3.3
	2,4'-DDT	0.10	3.3
	Hexachlorobenzene	0.10	3.3
	Oxychlordane	0.10	3.3

KATAHDIN ANALYTICAL SERVICES
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TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

Prepared By: Peter Lemay Date: 6/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/24/01

Operations Manager: John C. Bunton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Dennis F. Hoffman Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8151A	Format changes, added pollution prevention, other minor changes to sections 7 & 8 and QA Table	DN	1.24.01	1/31/01
02	Minor changes in sections 5 and 7	DN	4.9.02	4.9.02
03	Revised SOP to indicate Turbochrom is being used as instrument control and data collection software. Included Target related definitions. Changes to sections 7.5.3, 7.5.4, 7.6 and Table 1.	MRC	08.27.04	08.27.04
04	added Penta chloro phenol added alternating CV conc. (7.4.2) Table 1 - added CV conc. added Manual Int. SOP changed data review checklist	LAD	030405	030405
05	minor changes to reflect current practices, fix grammatical errors and formatting.	LAD	04/06	04/06

TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added waste stream to Sect. 1.0. Added N_2 gas as detector gas. Updated 2nd Column criteria; added flagging criteria. Changed correlation coefficient to Coefficient of determination in Sect. 7.2.1 and Table 1.	LAD	08/07	08/07
07	Section 8.3 - changed LCS and MS acceptance limits from nominal to statistical. Added DoD QSM limits for D. noseb. Table 1 - changed LCS acceptance criteria. Removed Table 4 - Nominal QC Acceptance Criteria	LAD	05/09	05/09
08	Updated sections 4.1, 7.2, 7.4, 7.5, 8.0, 10.0 and Table 1 for compliance with DoD QSM Version 4.1.	LAD	08/09	08/09
09	Sect. 2 - Removed reporting herbicides as acid equivalents. Sect. 8.2 - Updated values for Soil LCS and Surr. Spikes and Aq. Surr. spike. Table 2 - add DoD QSM QC criteria. Table 3 - Added method modification of not reporting herbicides as acid equivalents. Table 4 - Updated AQ and Soil PQLs. Updated Figures 1 and 2.	LAD	10/10	10/10
10	Section 7 - Added linear least squares and linear average calibration factor calibration models. Section 8 - Changed Corrective Action to non-conformance. Section 9. added MDL, LOQ and LOD information.	LAD	02/12	02/12
11	Sect. 7 - Changed reporting through Target to KIMS. Added Table 3 - DoD QSM 5.0 QC Requirements & renumbered subsequent Tables. Updated Fig.s 1 & 2. Changed KAS INC. to KAS throughout.	LAD	08/15	08/15
12	Sect. 5 - Added Standards to title. Sect. 7 - Added information for converting the methyl-ester back to free acid for reporting. Table 1 - Added linear calibration acceptance criteria. Table 3 - fixed symbols	LAD	08/16	08/16
13	Sect. 1, 8 and/or Table 1 - Added LLOQ definition and acceptence criteria. Clarified PQL, LOQ and LLOQ. Sect. 7 and Table 1 - Update CCV acceptance criteria to +/- 20%, added %Error and RSE calcs. Updated Project Review checklist.	LAD	06/17	06/17

TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect 1 & 4 – Added Chemstation, Sect. 4 – Updated column information. Sect. 7 - Updated run parameters, other minor changes. Sect 10 – Updated References Updated Figure 1	UAN	07/18	07/18
15	Sect. 7 – Changed RSD of CF criteria from 15% to 20%, added CCV concentration 0.25/25 ppm. Updated references. Other minor changes throughout.	UAN	02/20	02/20

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

1.0 SCOPE AND APPLICATION

This SOP details the procedure used by Katahdin Analytical Services personnel for the analysis of soil and water extracts for Herbicides, Method 8151. It is applicable to the following compounds: 2,4-D, 2,4,5-TP (Silvex), 2,4,5-T, 2,4-DB, Dalapon, Dinoseb, Dichloroprop, Dicamba, MCPA, and MCPP. Extracts are analyzed by Gas Chromatography-Electron Capture Detector. Detection limits achievable by this method are listed in Table 3.

The analyte Pentachlorophenol has also been analyzed for and quantitated using this method as part of a client's special request.

1.1 Definitions

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots the instrument response to the analyte versus concentration of known analyte standard.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

INDEPENDENT CALIBRATION VERIFICATION (ICV): A verification of the ratio of instrument response to analyte amount. ICV solutions are prepared from stock solutions which are independent from the stock solutions used to prepare the calibration standards.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix. For solid samples, a 1:2 (w/w) mixture of 400°C baked sand and sodium sulfate powder is used. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

AGILENT CHEMSTATION: A data acquisition system that is used to collect chromatographic data.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating

laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of herbicides by EPA Method 8151. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8151 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention and Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

2.0 SUMMARY OF METHOD

This SOP provides gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil and waste samples. Water samples are extracted with diethyl ether and then esterified with diazomethane. Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. The esters are analyzed by gas chromatography using an electron capture detector.

3.0 INTERFERENCES

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II or 6890 connected to the Chemstation data system, or equivalent.

4.1.2 Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-CLPesticides 30M x 0.53 mm x 0.5 um, RTX-CLPesticides2 30M x 0.53 mm x 0.42 um. Equivalent columns can be used.

4.1.3 Detectors Micro Electron Capture Detectors (Micro-ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Refrigerator for storage of extracts and standards.

5.0 REAGENTS AND STANDARDS

5.1 Solvent: Hexane - herbicide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like AccuStandard or other acceptable retailers as certified solutions of the methyl esters. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. The standard mix contains the following compounds: Dalapon, MCPP, Dicamba, MCPA, Dinoseb, Dichloroprop, 2,4-D, Silvex, 2,4,5-T, 2,4-DB and DCAA as the surrogate. Pentachlorophenol is also present in the standard mix.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in a separate logbook. The herbicide mix is diluted with hexane from concentrated stocks to give mixes at the following concentrations: 0.1/10, 0.25/25, 0.5/50, 0.75/75, 1.0/100, and 2.0/200 ug/ml. Note the compounds MCPA and MCPP are 100 times more concentrated than the other eight,

hence the ratio 0.1/10, etc. Pentachlorophenol is present in the standards at the lower of the two concentrations stated. Standards should be stored at 4°C.

5.2.3 Independent Calibration Verification Standard: Prepared at a mid point concentration using a standard independent of the calibration standards.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration at 4 (± 2) °C and analyzed within 40 days of extraction.

7.0 PROCEDURES

7.1 Instrument conditions

Refer to the instrument logbook for the current column and conditions.
Typical conditions are as follows:

- Column flow: 3.1 ml/min He
- Makeup flow: 60 ml/min Ar/Methane or Nitrogen
- Run time: 31 min
- Injector temp: 200
- Detector temp: 325
- Oven ramp: 100° (0) 8° /min - 200° (0) - 25° /min - 275° (4.9)
- Injector size: 2 ul split

7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard mix containing the analytes and surrogate listed in section 5.2.1 at the concentrations listed in section 5.2.2 is prepared.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard.

7.2.2 Linear calibration using the average calibration factor
The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD. If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.2.3 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.2.4 Non-linear calibration applying a second order polynomial (quadratic fit) equation

A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

NOTE: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear

calibration work originating in their state. In these cases, a linear calibration model must be used.

7.2.5 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\,\text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.2.6 The calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.2.7 The working calibration curve must be verified every 10 samples by injecting the 0.5/50 ppm or the 0.25/25 ppm calibration standard. If the response for any analyte varies from the expected response by more than ± 20%, a new calibration curve must be prepared for that analyte.

7.3 Retention time windows

7.3.1 Make three injections of all single component standard mixtures over the course of a 72 hour period.

7.3.2 The standard deviation of the three retention times is calculated for each single component standard.

7.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

7.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms.

7.4 Gas chromatographic analysis

7.4.1 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.

7.4.2 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.2 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 0.5/50ppm standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of $\pm$ 20%. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and a closing CV. The 0.25/25ppm standard should be used for the closing CV. If a second window of samples is run immediately after the closing CV, the concentration of the calibration standard at the completion of this window would be 0.5/50ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of a specific target analyte that exceeded the criterion.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >20%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.4.3 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.4.4 The identification of Herbicides is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows

established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.4.5 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.4.6 When a GC system is determined to be out of control because either a CV can not pass or a six point calibration does not meet the coefficient of determination criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, replacing the Y connector, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing an ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.

7.4.7 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

7.4.8 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.4.8.1 Water: Concentration (ug/L) = (C) (Vt)/(Vs)

7.4.8.2 Soil/Sediment:Concentration (mg/kg) = (C) (Vt) / ((Ws) (D))

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.4.9 The final concentration is calculated as the methyl ester form of the herbicide. This must be adjusted to account for the molecular weight difference of the methyl ester form and the free acid form of the herbicide. The calculation is final concentration (in ug/Kg or ug/L) * MW free acid/ MW methyl ester.

This calculation is done after the data has been uploaded into KIMs for final reporting. The final amount on the quantitation reports will differ slightly from the amounts on the Report of Analysis

7.5 Data Review

7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.6.

7.5.2 Surrogate recovery

All recoveries must meet the most recently established laboratory acceptance limits.
The sample is evaluated for the recovery of the surrogate. If the surrogate recovery is high and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recovery is low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recovery is low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recovery of the surrogate is low or high, and the blank does not contain any target analytes above the PQL, and the recovery of the surrogate in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes

above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of the surrogate and the analyte spikes are low, the samples may need to be reextracted.

7.5.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to the current revision of Katahdin SOP QA-812 “Manual Integration on GC/MS, GC, HPLC and IC Datasystems” for more information.

7.5.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value. The higher of the two concentrations is reported unless matrix interference is causing

erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target.

Reasons to consider a detection may be a false positive:

- an analyte is present on one column but its concentration is below the PQL,
- an analyte is present on one column but does not confirm on the other channel,
- an analyte is present on both columns but the concentrations differ by more than 40%
- an analyte is present but its retention time is ± 0.04 minutes or more than the retention time of the analyte in the preceding CV.

7.6 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMs. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may

rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.1.1 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.1.2 LCS, MS/MSD and Surrogate Spike concentrations:

The LCS and the MS/MSD are spiked with the ten single component herbicides at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS mg/Kg
All Herbicides except	5.0	167
MCPA and MCPP	500	16700

The surrogate spike concentration in the final extracts is:

	WATER ug/L	SOILS mg/Kg
2,4-Dichlrophenylacetic acid (DCAA)	5.0	167

8.1.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For DoD work, the recoveries are compared to DoD QSM acceptance limits.

Dinoseb in a soil matrix recovers poorly. SW846 Method 8151 states that the soil hydrolysis step may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids. Dinseb is also listed as a poor performer in the Department of Defense Quality Assurance Manual, current revision. For these reasons Katahdin Analytical Services defaults to the DoD QSM 4.2 limits of 5-130% for this compound.

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.
DoD work requires Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.1.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.2 Non-conformance report (NCR): Whenever data is not acceptable because of a failing LCS or surrogate recovery, a NCR must be initiated as soon as possible.

8.3 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8151 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 8151A, Revision 1, December 1996, Final Update III to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

LIST OF TABLES AND FIGURES

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL / LLOQ	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: ie. if the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	Laboratory statistically derived limits Dinoseb in soil – DoD QSM 4.2 limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CV	One after every 10 samples: alternating between 0.5/50ppm and 0.25/25ppm concentration	± 20% D	(1) Evaluate the samples: If the %D > +20% and sample results are < PQL, narrate. If %D > ± 120% only on one channel, narrate. If %D > ± 20% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: ie. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate	One sample duplicate per ten samples if requested	RPD ≤20	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
5 or 6pt calibration of Herbicide Mix	Initial cal prior to sample analysis	Linear: %RSD ≤ 20% or (r) ≥ 0.995 Non-linear: (r^2) ≥ 0.990 % Error ≤ 30% or RSE ≤ 20% (or ≤30% for poor performers)	(1) Repeat Initial calibration .
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
MDL study, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 2

DoD QSM 5.0 / 5.1 / 5.3 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 2

DoD QSM 5.0 / 5.1 / 5.3 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 2

DoD QSM 5.0 / 5.1 / 5.3 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-305-15	METHOD EPA 8151, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	7.5.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms.	7.5.2.1 Plus or minus three times the standard deviation of the retention times for each standard will be used to define the retention time window. 7.5.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 4

PQLS FOR METHOD SW8151

Parameter/Method	Analyte	Practical Quantitation Limit (PQL)	
		(ug/L)	(ug/kg)
Herbicide/	2,4-D	3.0	67
SW846 8151	2,4,5-TP	1.0	33
	2,4-DB	3.0	33
	2,4,5-T	3.0	33
	Dalapon	5.0	170
	Dicamba	5.0	33
	Dichloroprop	3.0	67
	Dinoseb	5.0	170
	MCPA	150	5000
	MCPP	100	3300

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services
GC Laboratory Instrument Runlog
Instrument: GC08
Amount Injected: 2 uL
Column Numbers: 478/479
Method: SW846 8081 / 8082 / 8151 / 8011
(circle) EPA 504.1 / 556 / 608

Hexane Lot #:	DT241
Standard	Standard ID
Herb 0.5	PQ063
↓ 0.25	PQ068

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
4-17-18	BF	SLDOC146	Herb 0.5	Y	WG226453-7	Herb168	
		147	0.1		-2		PQ067
		148	0.25		-3		
		149	0.75		-4		PQ065
		150	1.0		-5		PQ066
		151	2.0		-6		PQ064
		152	Herb IND	↓	-7		PQ090
		153	WG226342-1 SSTC	Y			
		154	-2				
		155	SL2813-1				
		156	SL2964-1				
		157	SL3034-1	↓			
↓	↓	158	Herb 0.5	Y	-8	↓	
4-17-18	BF	SLDOC159	PRIME	N	WG226651	Herb168	
		160	Herb 0.5 IND	Y			
		161	WG226604-1	Y			
		162	-2				Dicamba168
		163	SL3069-4				
		164	-5				
		165	-6				
		166	SL3104-7				
		167	-8				
		168	-9				
		169	-10				
		170	-12				
		171	SL3151-20	↓			
		172	RINSE	N			Dark samples
		173	Herb 0.25	Y			
4-18-18	BF	SLDOC174	PRIME	N	WG226714	Herb168	fine current
		175	Herb 0.5	Y	-1		

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QAGC373

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TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

FIGURE 2

REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Int.*_____ ,) **DueDate**_____.

Client:	**Verbal Review**	**Primary Review**	**Secondary Review**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT* ***ND's to*** - ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitaion Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐) .	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement _Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD ○ LAB ○	☐ FORM 6 (s) DOD ○ LAB ○
☐ FORM 4 (s)	☐ FORM 7 (s) DOD ○ LAB ○
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ ○ MDL ○ LOD ○
☐ FORM 8 (s)	☐ Flagging **B** ○ **L** ○ **M** ○ **C** ○
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ ○ MDL ○ LOD ○	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD ○ LAB ○	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD ○ LAB ○	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	Exceedences
< 11	0
11 to 30	1
31 to 50	2
51 to 70	3
71 to 90	4
> 90	5

QA-044 – Revision10 – 04/07/2017

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 2/15/01

Operations Manager: John C. Burton Date: 2/14/01

QA Officer: Deborah J. Nadeau Date: 2.14.01

General Manager: Donald F. Hutchins Date: 2/14/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, other updated changes to all sections.	DN	2.14.01	2/14/01
02	Minor changes to sections 5,6 & 7. Grammatical errors corrected	DN	4.9.02	4.9.02
03	added definitions modified Sections 5,7,8 and 9 modified table to include extra cmpds	LAD	01-31-05	01-31-05
04	Added pollution control and waste disposal to sect. 1A. Added kims definition to sect. 1.1 and 7.9.1. Removed all references to 60mL vials. Edited AQ LCS prep. minor changes throughout to reflect current practice. Fixed numbering and formatting	LAD	09/07	09/07
05	Corrected references in text to Tables 1,3 & 4. Reworded Sect.s 8.14.4, 8.1.5, 8.4.2.3 and 8.4.3 for clarity. Changed Lab fortified blank to LCS and lab fortified Matrix to MS	LAD	03/08	03/08

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Removed references to obsolete method CA - 320 (method 5030)	LAD	09/08	09/08
07	Sect. 7: Added calibration procedure using CFs and % RSD, removed calibration procedure using linear regression, Corrected soil calculation. Sect. 8: Clarified MS/D frequency. Removed annual RL verification and added non-conformance report criteria. Sect 9 & 10: Added references	LAD	04/10	04/10
08	Added ICV criteria. Updated Sections 5.5, 5.6, 7.1, 8.4 and Table 3 with new stock concentrations, amounts added, and final concentrations for aqueous and soil LCSs and MSs Changed ICAL table to include lower calibration level. Table 5 - updated Benzene PQL to 4 ug/L. Added linear calibration criteria.	LAD	12/11	12/11
09	Sect. 1 - Added Chemstation definition. Sect. 5 & 8 - Corrected MS solution vs. stock solution. Sect. 7 - Changed reporting through Quickforms to KIMS. Table 1 - Added linear regression criteria. Updated Fig. 1 & 2.	LAD	04/13	04/13
10	Sect. 5 - Edited for clarification. Sect. 7 - Corrected closing CCV criteria, removed baseline correction reference. Sect. 8 - removed naphth. exception, reword NCR information. Table 5 - updated Benzene PQL. Updated Fig. 2.	LAD	08/15	08/15
11	Sect. 5.0 - updated title. Sect. 10 - updated method references. Added OI Analytical system procedures throughout. updated Table 3, 5 and Figure 2.	LAD	09/17	09/17

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

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TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

1.0 SCOPE AND APPLICATION

This method is designed to measure the collective concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. Volatile aliphatic hydrocarbons are collectively quantitated within two ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantitated within the C_9 to C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 36°C and 220°C.

This method is based on a purge-and-trap, gas chromatography (GC) procedure using in-series Photoionization and Flame Ionization Detectors (PID/FID). This method should be used by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

This method is also able to measure the individual concentrations of the VPH Target Analytes benzene, toluene, ethylbenzene, xylenes (BTEX), naphthalene, and methyl-tert-butylether (MTBE) in water and soil. Use of this method to identify and quantitate these Target Analytes is optional.

Petroleum products suitable for evaluation by this method include gasoline, mineral spirits, and certain petroleum naphthas. This method, in and of itself, is not suitable for the evaluation of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products that contain a significant percentage of hydrocarbons heavier than C_{12}.

This method includes a series of data manipulation steps to determine the concentrations of aliphatic and aromatic ranges of interest.

Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target Analyte. Confirmatory analysis by a GC/MS procedure or other suitable method is recommended in cases where a Target Analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where co-elution of a non-targeted hydrocarbon compound is suspected.

This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance. MADEP encourages methodological innovations which (a) better achieve method and/or data quality objectives, (b) increase analytical precision and accuracy, (c) reduce analytical uncertainties and expenses, and/or (d) reduce the use of toxic solvents and generation of hazardous wastes. Laboratories that modify this method must achieve all required performance and acceptance standards, and must have on file a Standard Operating Procedure which thoroughly describes the revised

or alternative method and documentation which demonstrates an equivalent or superior level of performance. All significant modifications to the method must be disclosed and described on the data report form.

1.1 Definitions

Analytical Batch is defined as a group of field samples with similar matrices which are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

Calibration Check Standard is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

Calibration Standards are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

C_5 through C_8 Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram from n-pentane (C_5) to just before n-nonane (C_9).

C_9 through C_{12} Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram from n-nonane (C_9) to just before naphthalene.

C_9 through C_{10} Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds which elute on the PID chromatogram from just after o-xylene to just before naphthalene. Although it is an aromatic compound with 10 carbon atoms, naphthalene is excluded from this range because it is evaluated as a separate (Target) analyte.

Field Duplicates are defined as two separate samples collected at the same time and place under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

Laboratory Control Sample (LCS) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution.

Laboratory Duplicates are defined as split samples taken from the same sampling container and analyzed separately with identical procedures. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

Matrix Spike (MS) Sample is defined as an environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The MS sample is treated and analyzed exactly as a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the MS sample corrected for background concentrations.

Laboratory Method Blank (LMB) is defined as an aliquot of laboratory reagent grade water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

Matrix Spiking Solution is defined as a solution prepared independently from the calibration standards, containing known concentrations of method analytes.

System Solvent Blank is defined as an aliquot of method solvent (e.g., methanol) that is directly purged into the GC system. The purpose of the Solvent Blank is to determine the level of noise and baseline rise attributable solely to the GC system, in the absence of any other analytes or contaminants.

Target VPH Analytes are defined as benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, naphthalene, and methyl-tert-butylether.

Unadjusted C_5 through C_8 Aliphatic Hydrocarbons are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-pentane (C_5) to just before n-nonane (C_9).

Unadjusted C_9 through C_{12} Aliphatic Hydrocarbons are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-nonane (C_9) to just before naphthalene.

Volatile Petroleum Hydrocarbons (VPH) are defined as collective fractions of hydrocarbon compounds eluting from n-pentane to naphthalene, excluding Target VPH Analytes. VPH is comprised of C_5 through C_8 Aliphatic Hydrocarbons, C_9 through C_{12} Aliphatic Hydrocarbons, and C_9 through C_{10} Aromatic Hydrocarbons.

Volatile Petroleum Hydrocarbon (VPH) Component Standard is defined as a 15 component mixture of the aliphatic and aromatic compounds listed in Table 3. The compounds comprising the VPH Component Standard are used to (a) define the individual retention times and chromatographic response factors for each of the Target VPH Analytes, (b) define and establish the windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and (c) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing laboratory status reports, and generating final reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: Data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of VPH by MADEP VPH-04-1.1. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of VPH by MADEP VPH-04-1.1 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP. Purge vial and methanol waste are disposed of in the “A” waste satellite accumulation area located between GC02 and GC09.

2.0 SUMMARY OF METHOD

2.1 Samples are analyzed using purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic

compounds. Detection is achieved by a Photoionization detector (PID) and flame ionization detector (FID) in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations of targeted analytes and collective concentration of aromatic hydrocarbons within the C_9 through C_{10} range. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C_5 through C_8 and C_9 through C_{12} ranges.

2.2 This method is suitable for the analysis of waters, soils, and sediments. Water samples may be analyzed directly for volatile petroleum hydrocarbons by purge-and-trap concentration and gas chromatography. Soil samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is then analyzed by purge-and-trap GC.

2.3 This method is based on (1) USEPA Methods 5030, 8000, 8020, and 8015, SW-846, "Test Methods for Evaluating Solid Wastes", 3rd Edition, 1986; (2) Draft "Method for Determination of Gasoline Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Gasoline Range Organics", Wisconsin Department of Natural Resources, PUBL-SW140, 1992.

3.0 INTERFERENCES

3.1 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage or by dissolution of volatiles into the methanol used for preservation. Trip blanks prepared from both laboratory reagent grade water and methanol should be carried through sampling and subsequent storage and handling to serve as a check on such contamination.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with laboratory reagent grade water or solvent. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling-point compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in an oven at 105°C between analyses. The trap and other parts of the system are also subject to contamination, therefore, frequent bake-out and purging of the entire system may be required. A screening step is recommended to protect analytical instrumentation. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of a system solvent blank or laboratory method blank to check for cross-contamination.

3.3 Certain organic compounds not associated with releases of petroleum products, including chlorinated solvents, ketones, and ethers, will be quantitated as Volatile Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.

3.4 The response selectivity of a photoionization detector (PID) is used in this method to differentiate aromatic hydrocarbons from aliphatic hydrocarbons. All compounds eluting on the PID chromatogram after o-xylene are identified by the method as aromatic hydrocarbons. This will lead to an overestimation of aromatic hydrocarbons within samples, as certain aliphatic compounds will elicit a response on the PID, particularly unsaturated compounds such as alkenes. The significance and implications of this overestimation will vary from sample to sample; where less conservative data are desired, additional actions should be considered to minimize the detection of non-aromatic compounds, including the use of a lower energy PID lamp, different chromatographic columns, and/or addition of a pre-analysis sample cleanup step.

4.0 APPARATUS AND MATERIALS

4.1 The following glassware is used in this method:

4.1.1 VOC Vials: 40 mL VOC vials with Teflon/ silicone septa for waters and soils.

4.1.2 Class “A” Volumetric flasks: 10 mL, 50 mL, 100 mL, and 1,000 mL with a ground-glass stopper.

4.1.3 Disposable pipettes: Pasteur.

4.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples.

4.3 Gas Chromatography

4.3.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph and purge-and-trap concentrator. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

4.3.2 Chromatographic Column: The required column is: 105M x 0.53 mm I.D. Restek RTX 502.2 with 3 micron film thickness, or column with equivalent chromatographic properties.

NOTE: Based upon data obtained from the Round Robin testing programs, the choice of chromatographic column may have a significant impact on the apportionment and quantitation of aliphatic and aromatic compounds within the fractional ranges specified in this method. Substitution of the required column is not allowed, unless it can be demonstrated that the selected column has equivalent chromatographic properties and retention times for the aliphatic and aromatic compounds and ranges of interest. To demonstrate equivalency of column chromatography, a neat gasoline standard must be analyzed on both the required column and the proposed substitute column, with all other run and system parameters held constant. The concentrations of C_5-C_8 and C_9-C_{12} Aliphatic Hydrocarbons must be determined for each column (in which the concentration of the Target/aromatic analytes have been subtracted from the GC/FID response). The Relative Percent Difference between the concentrations of each fraction obtained for each column must be equal to or less than 25%.

4.3.3 Detectors: The method requires the use of a Photoionization Detector (PID) in series with a Flame Ionization Detector (FID); the PID first in the series. The method is based upon the use of a 10.0 +/- eV PID lamp, although lower energy lamps are permissible in order to minimize PID response to aliphatic compounds. In lieu of an in-series arrangement, in-parallel PID and FID units may be also used.

4.3.4 Purge-and-trap device: The purge-and-trap device consists of a sample purger, a trap, and a desorber. Several complete devices are commercially available.

4.3.4.1 The purging chamber must be designed to accept 5 mL samples with a water column at least 3 cm deep. Purging devices larger than 5 mL have a reduced purging efficiency and should not be used. The gaseous headspace between the water column and the top of the vessel should be at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. Fritted glass or needle sparge cells may be used. If needle sparge cells are used, the purge gas must be introduced no more than 5 mm from the base of the water column. Alternate sample

purge devices may be used, provided equivalent performance is demonstrated.

4.3.4.2 The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inches. The trap should be packed with 400 mg of Carbopack B (Supelco Cat. No. 209273). Alternative trap packing materials include: Tenax GC (or equivalent); 7.6 cm Carbopack B and 1.3 cm Carbosieve S-III (Supelco Cat No. 2-0321); 7 cm Carbopack C and 1.2 cm Carbopack B (Supelco Cat No. 2-1064); or equal volumes of Tenax, silica gel, and charcoal as described in EPA SW-846 Method 5030. In general, Carbopack trap packing materials are recommended because they have less of a tendency to retain methanol, which could interfere with the elution of pentane and quench the FID flame. The trap length and packing materials may be varied as long as equivalent performance has been verified.

4.3.4.3 Prior to initial use, the Carbopack B trap should be conditioned overnight at 270°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to a hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min. at 260°C with backflushing. The trap may be vented to the analytical column during daily conditioning, however, the column must be run through the temperature program prior to analysis of samples. Devices other than the traps recommended in Section 4.3.4.2 should be conditioned and desorbed according to the manufacturer's guidelines.

4.3.4.4 The desorber should be capable of rapidly heating the trap to 240°C for desorption.

4.4 Data System: A data system which allows the continuous acquisition of data throughout the duration of the chromatographic program must be interfaced to the GC. The data system must be capable of storing and re-integrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. All data editing will be reviewed by the Department Manager or qualified designee before samples are reported.

4.5 Ultrasonic bath.

4.6 Syringes: 5 mL Luerlock glass hypodermic and 5 mL gas-tight syringe with shutoff valve.

4.7 Syringe valve: Two-way, with luer ends.

4.8 Microsyringes: 1 µL, 5 µL, 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL.

4.9 Spatula: Stainless steel.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

5.1.1 Laboratory Reagent Grade Water: Organic-free water

5.1.2 Methanol: purge-and-trap grade or equivalent; store away from other solvents to prevent cross-contamination

5.1.3 Organic-free sand: sand baked in an oven at 400°C for four hours to remove organic contaminants

5.2 Stock Standard Solution

5.2.1 The stock standard solution consists of the 15 VPH component standards and a surrogate standard that is purchased from vendors like ULTRA Scientific at a concentration of 10000 µg/mL. The solution is stored at -10°C to -20°C and protected from light.

5.2.2 The stock standard solution must be replaced after six months from the date of opening or sooner if the manufacture's date is less.

5.3 Primary Dilution Standard

5.3.1 The primary dilution standards are prepared in methanol at concentrations listed in Table 3. These standards are stored at -10°C to -20°C.

5.3.2 The primary dilution standards should be replaced at least monthly.

5.4 VPH Calibration Standards

VPH calibration standards are prepared by injecting an aliquot of either the primary dilution standard or the stock standard directly into a 5 mL syringe if using the Tekmar system or a 50 mL volumetric flask if using the OI Analytical system. The volumes added are listed in Table 3.

5.5 Surrogate Standard

5.5.1 The analyst must monitor both the performance of the analytical system and the effectiveness of the method in dealing with sample matrices by spiking each sample, blank, and matrix spike with a surrogate standard. The surrogate standard is also added to the VPH calibration standard solutions. The recommended surrogate standard is 2,5-dibromotoluene, which elutes after all aliphatic and aromatic compounds of interest. The use of another surrogate compound is permissible.

5.5.2 Surrogate Spiking Solution

5.5.2.1 For aqueous samples: Prepare a surrogate spiking solution at 100 µg/mL in 1.0 mL of methanol from the 5000 µg/mL stock standard solution. Add 5 µL of this surrogate spiking solution directly into the 5 mL syringe with every aqueous sample and method blank if using the Tekmar system. Add 50 µL of this surrogate spiking solution if using the OI Analytical system. This will correlate to a concentration of 100 µg/L in the sample.

5.5.2.2 For soil samples: Add 70 uL of the 5000 µg/mL stock standard solution to every soil sample and method blank during the extraction step. This will correlate to a concentration of 93.3 µg/L in the sample for a 15 g extraction.

5.6 Matrix Spiking Solution

5.6.1 The matrix spiking solution consists of the Targeted VPH analytes and the surrogate.

5.6.1.1 For aqueous samples: Prepare a matrix spiking solution at 100 µg/mL in 1.0 mL of methanol from the 5000 µg/mL stock standard solution. Add 5 µL of this matrix spiking solution directly into the 5 mL syringe with every aqueous laboratory control sample (LCS) and matrix spike sample (MS) if using the Tekmar system. Add 50 µL of this matrix spiking solution directly into the 50 mL volumetric flask with every aqueous laboratory control sample (LCS) and matrix spike sample (MS) if using the OI Analytical system. This will correlate to a concentration of 100 µg/L in the sample.

5.6.1.2 For soil samples: Add 35 µL of the 10000 µg/mL stock standard solution to every LCS and MS during the extraction step.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous Samples

6.1.1 Aqueous samples should be collected in duplicate (or the number of vials directed by the laboratory) without agitation and without headspace in contaminant-free glass VOC vials with Teflon-lined septa screw caps. The Teflon liner must contact the sample. Samples must be acidified to a pH of 2 or less at the time of collection. This can generally be accomplished by adding 3 or 4 drops (0.1 to 0.2 mL) of 1:1 HCl (1 part laboratory reagent grade water and 1 part concentrated HCl) to a 40 mL sample vial. Samples must be cooled to 4°C immediately after collection.

6.1.2 A chain of custody form must accompany all sampling vials and must document the date and time of sample collection and acid preservation. The pH of all water samples must be determined by the laboratory unless sample vials containing acid for field preservation were supplied by the laboratory (this must be noted on the chain of custody). The pH measurement may be performed on left over sample. Any sample found to contain a pH above 2 must be so noted on the laboratory/data report sheet.

6.1.3 A laboratory reagent grade water trip blank should accompany each batch of water samples.

6.1.4 Any sample received by the laboratory that is not packed in ice or cooled to 4°C must be so noted on the laboratory/data report sheet.

6.1.5 Aqueous samples must be analyzed within 14 days of collection.

6.2 Soil Samples

6.2.1 Soil samples must be collected in a manner that minimizes sample handling and agitation. The use of specially designed air-tight collection samplers or a 30 mL plastic syringe with the end sliced off is recommended. All sediment must be removed from the glass threads of the vial to ensure an adequate seal. Samples must be cooled to 4°C immediately after collection.

6.2.2 **Methanol preservation of soil samples is mandatory.** Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air-tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection. Additional details and

recommendations on soil sampling may be found in method MADEP VPH-04-1.1.

6.2.3 The desired ratio of methanol-to-soil is 1 mL methanol/1 gram soil, ± 25%. The exact weight of the soil sample and volume of methanol must be known or ascertained by the laboratory when calculating and reporting soil concentration data. A recommended practice is for a laboratory to provide to a field sampling technician labeled, pre-weighed sampling vials with a measured volume of methanol, and a scribed mark indicating the level of methanol that should exist in the vial when the required quantity of soil sample has been added. This requires an estimate on the density and moisture content of the soil sample; a good estimate for most soils is 8-10 mL of displaced volume for 15 grams soil. **In all cases, the soil sample in the vial must be completely covered by methanol.**

6.2.4 Samples for VPH analysis should be collected in duplicate 40 mL vials. An additional sample of the soil must also be obtained (without methanol) to allow for a determination of soil moisture content and VPH dry weight correction factors.

6.2.5 A methanol trip blank should accompany each batch of soil samples.

6.2.6 A chain of custody form must accompany all sampling vials and must document the date and time of sample collection and, where appropriate, the volume of methanol added. Observations of vial leakage must be so noted on the laboratory/data report sheet.

6.2.7 Soil samples must be analyzed within 28 days of collection.

6.3 A summary of sample collection, preservation and holding times is provided in Table 4.

7.0 PROCEDURES

7.1 Sample Preparation and Purging

7.1.1 It is highly recommended that all samples be screened prior to analysis. This screening step may be analysis of a soil sample's methanol extract (diluted) or the hexadecane extraction and screening method (SW-846 Method 3820).

7.1.2 Water Samples - Introduce volatile compounds into the gas chromatograph using a purge-and-trap concentrator.

7.1.2.1 If using the Tekmar system remove the plunger from a 5 mL syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample and vent any residual air while adjusting the sample volume to 5 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if a second analysis is needed an alternate vial will be used. If an alternate vial is not available then the compromised vial will be used and noted.

7.1.2.2 If using the OI Analytical system, place the sample VOA vial into the OI Analytical sample tray and sequence for analysis. The OI Analytical unit will automatically transfer the sample to the sparge vessel.

7.1.2.3 If necessary, samples should be diluted prior to injection into the purge chamber. In such cases, all steps must be performed without delay until the diluted sample is in a gas-tight syringe.

7.1.2.3.1 Dilutions may be made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for highly concentrated samples.

7.1.2.3.2 Calculate the approximate volume of laboratory reagent grade water to be added to the volumetric flask selected and add slightly less than this volume of laboratory reagent grade water to the flask.

7.1.2.3.3 Inject the proper aliquot of sample from the syringe prepared in Paragraph 7.1.2.1 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with laboratory reagent grade water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions. Alternatively the dilutions can be made directly in the glass syringe to avoid further loss of volatiles.

7.1.2.3.4 Fill a 5 mL syringe with diluted sample as in Paragraph 7.1.2.1.

7.1.2.3.5 Add 5 µL of the surrogate spiking solution through the valve bore of the syringe.

7.1.2.3.6 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.

7.1.2.3.7 Close the valve and start the LSC 3000 sample concentrator. Refer to the instrument maintenance logbook for the current purge-and-trap operating parameters.

7.1.2.3.8 If the concentration of an analyte or hydrocarbon fraction in a sample exceeds the calibration range, a dilution of the sample is required. If a sample analysis results in a saturated detector response for a compound, the analysis must be followed by a blank laboratory reagent grade water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.1.2.3.9 All dilutions should keep the detector response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.1.2.4 If using the OI Analytical system to dilute a sample use the steps in Paragraphs 7.1.2.3.1 through 7.1.2.3.3 to achieve proper dilution.

7.1.3 Soil/Sediments - Soil and sediment samples are extracted with methanol. An aliquot of the extract is added to laboratory reagent grade water and introduced into the gas chromatograph using a purge-and-trap concentrator.

7.1.3.1 Weigh the sample vial to 0.1 g in a top loading balance and determine the weight of the soil/sediment sample; this determination requires knowledge of the empty/tarred weight of the sample vial and volume/weight of methanol preservative that was added to the sample vial.

7.1.3.2 Add 70 uL of the surrogate spiking solution. The concentration and/or volume of the surrogate spiking compound may need to be increased for samples that are highly contaminated (based upon screening and/or visual/olfactory evidence), to prevent dilution to

below detectable limits. Shake the sample for 2 minutes and sonicate for 20 minutes.

7.1.3.3 Allow sediment to settle until a layer of methanol is apparent.

7.1.3.4 Using a microliter syringe, withdraw a 20 µL aliquot of the methanol extract for sparging if using the Tekmar system. Withdraw a 200 µL aliquot of the methanol extract for sparging if using the OI Analytical system. Sample screening data can be used to determine the volume of methanol extract to add to the 5 mL of laboratory reagent grade water for analysis. All dilutions must keep the response of the major constituents in the upper half of the linear range of the calibration curve.

7.1.3.5 If using the Tekmar system remove the plunger from one 5 mL Luerlock type syringe and fill until overflowing with laboratory reagent grade water. Replace the plunger and compress the water to vent trapped air. Pull the plunger to 5 mL for addition of the sample extract. Add the volume of methanol extract (20 µL maximum). Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.

7.1.3.6 If using the OI Analytical system remove the glass stopper from a 50 mL volumetric flask and fill up to the mark with laboratory reagent grade water. Add the volume of methanol extract (200 µL maximum) into the volumetric flask. Cap the flask, invert, and shake three times. Carefully pour the contents of the flask into a 40 mL VOA vial until the vial is overflowing and be sure there is no head space present. After prepping the VOA vial place the sample into the OI Analytical sample tray and sequence for analysis. The OI Analytical unit will automatically transfer the sample to the sparge vessel.

7.1.3.7 If the responses exceed the calibration or linear range of the system, use a smaller aliquot of methanol.

7.2 GC Conditions

7.2.1 Refer to instrument maintenance logbook for the current oven conditions. The typical oven temperature program is: Oven temperature 35°C, hold for 8 min, then to 90°C at 3°C/min, to 140°C at 5°C/min, to 230°C at 45°C/min; hold for 10.5 min. Conditions may be altered to improve resolution of volatile petroleum hydrocarbons.

7.2.2 Gas Flows: The recommended carrier gas is helium.

7.2.2.1 Carrier gas flow: 10 mL/min.

7.2.2.2 Air: 160 mL/min

7.2.2.3 Hydrogen: 30 mL/min

7.2.2.4 Make up gas flow: 15 mL/min

7.2.3 Miscellaneous:

7.2.3.1 FID temperature: 220°C

7.2.3.2 PID temperature: 220°C

7.2.3.3 Injection port temperature: 200°C

7.2.3.4 Column head pressure: 30 psi

7.3 Retention Time Windows

7.3.1 Before establishing retention time windows, make sure the GC system is within optimum operating conditions. Make three injections of the VPH Component Standard throughout the course of a 72 hr period. Serial injections over less than a 72 hr period may result in retention time windows that are too tight.

7.3.2 Calculate the standard deviation of the three absolute retention times for each individual compound in the VPH Component Standard.

7.3.3 The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each standard. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.3.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.

7.3.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the lab.

7.3.6 VPH retention time (Rt) windows of the aliphatic ranges are defined as beginning 0.1 minutes before the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound, except for C_9, which is both a beginning and ending marker compound for two different ranges.

The C_5-C_8 Aliphatic Hydrocarbon range ends immediately (0.1 min) before the elution of the n-C_9 peak. The C_9-C_{12} Aliphatic Hydrocarbon range begins 0.1 min before the elution of n-C_9, therefore there is no overlap of the two ranges and the n-C_9 peak is only included in the C_9-C_{12} Aliphatic Hydrocarbon range.

The VPH retention time (Rt) window for the C_9-C_{10} Aromatic Hydrocarbons is defined as beginning 0.1 minutes after the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound, VPH marker compounds and windows are summarized below.

VPH Marker Compounds

Hydrocarbon Range	Beginning Marker Compound	Ending Marker Compound
C_5 - C_8 Aliphatic Hydrocarbons (FID)	0.1 min before Pentane	0.1 min before n-Nonane
C_9 - C_{12} Aliphatic Hydrocarbons (FID)	0.1 min before n-Nonane	0.1 min before Naphthalene[1]
C_9 -C_{10} Aromatic Hydrocarbons (PID)	0.1 min after o-xylene	0.1 min before Naphthalene[1]

[1]The retention time for Dodecane (C_{12}) is approximately 2 minutes less than the retention time for naphthalene, using the column and chromatographic conditions recommended for this method. For simplicity, naphthalene is used as the ending marker for the C_9-C_{12} Aliphatic Hydrocarbon range.

7.4 External Standard Calibration Procedure

7.1.3 The average calibration factor procedure

Analyze each of the six VPH Calibration standards following the procedure outlined in section 7.5. Tabulate the area responses against the concentration injected. The ratio of area response to the concentration injected, defined as the calibration factor (CF), may be calculated for target VPH analytes using the equation below.

Calibration Factor (CF) = area of peak/concentration purged (µg/L)

The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest, as determined using the equation below. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.

%RSD = (stand dev of 6 CFs/mean of 6 CFs) x 100

A collective calibration factor must also be established for each hydrocarbon range of interest: C_5-C_8 Aliphatic Hydrocarbons, C_9-C_{12} Aliphatic Hydrocarbons and the C_9-C_{10} Aromatic Hydrocarbons. Calculate the collective CFs for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CFs for the C_9-C_{10} Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. Refer to Table 3 for the concentrations of the ranges. The results can be used to calculate the ratio of the peak area response summation to the concentration injected, defined as the CF, for the hydrocarbon ranges using the equation below. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest, as determined using the equation above.

CF = area summation of range components/total conc. purged (µg/L)

7.4.2. Linear Regression

Analyze each of the six VPH Calibration standards following the procedure outlined in section 7.5. Tabulate the area responses against the concentration injected. A linear calibration applying a first order equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient (r) must be greater than or equal to 0.990. The equation is:

$y = mx + b$

where: y = Instrument response
m = Slope of the line
x = Concentration of the calibration standard or range
b = The intercept

Calculate a linear regression (LR) for the individual VPH target analytes and surrogate.

A collective calibration curve must also be established for each hydrocarbon range of interest: C_5-C_8 Aliphatic Hydrocarbons, C_9-C_{12} Aliphatic Hydrocarbons and the C_9-C_{10} Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. Refer to Table 3 for the concentrations of the ranges.

If linear regression is used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.

7.4.3 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, and surrogates at mid-calibration range concentration and is obtained from a source independent of the initial calibration source. Please refer to Table 1 for acceptance criteria and corrective action for this standard. When analyzed after an initial calibration and directly before samples, this standard also doubles as an LCS sample.

7.4.3.1. Target VPH Analytes and C_9 to C_{10} Aromatic Hydrocarbons are quantitated on the PID chromatogram.

7.4.3.2. C_5 through C_8 and C_9 through C_{12} Aliphatic Hydrocarbons are quantitated on the FID chromatogram.

7.4.4 At a minimum, the working calibration curve or calibration factor must be verified on each working day, after every 20 samples and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than ± 25%, as determined using the equation below, a new calibration curve must be prepared for that analyte. Greater differences are permissible for n-nonane (±30%).

Linear Calibration Percent Difference (%D)

$\%D = (R_1 / R_2) \times 100$

where: R1 = Calculated concentration from curve.
R2 = Expected concentration.

Average Calibration Factor Percent Difference (%D)

%D = (CFavg – CFcc)/(CFavg)x100

where: CFavg = Average calibration factor calculated from initial calibration
CFcc = Calibration factor calculated from continuing calibration

N-nonane has an allowed %D of ≤ 30%; if the %D exceeds this value, it may be narrated without the necessity of recalibration.

7.5 GC Analysis

7.5.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by samples interspersed with blanks and QC samples. The sequence ends when the set of samples has been analyzed or when qualitative and/or quantitative QC criteria are exceeded.

7.5.2 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.

7.5.2.1 Tentative identification of an analyte occurs when a peak from a sample falls within the daily retention time window. Confirmation on a second GC column or by GC/MS analysis may be necessary.

7.5.2.2 Coelution of the m- and p- Xylene isomers may occur.

7.5.2.3 Validation of GC system qualitative performance must be accomplished by the analysis of mid-level standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

7.5.3 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peak elutions between specified range "marker" compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual check be initially performed, to document proper integration functions.

7.5.4 **When quantifying on a peak area basis by external calibration, collective peak area integration for the fractional ranges must be from baseline-to-baseline (i.e. must include the unresolved complex mixture "hump" areas).** For the integration of individual Target Analytes, surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.

7.5.5 Baseline correction using a system solvent blank is permissible, if conducted in accordance with the procedures and requirements specified in Section 7.7.4.

7.5.6 If the VPH Target Analytes are to be quantitated using this method, and the response for an individual analyte exceeds the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale.

7.5.7 For non-target analytes and target analytes eluting in the aliphatic or aromatic fractions, the upper linear range of the system is based on the highest calibration standard. Refer to Table 3 for concentrations.

7.6 Calculations (external standard)

The concentration of targeted analytes and hydrocarbon ranges in a sample may be determined by calculating the amount of analyte or hydrocarbon range purged, from the peak response using the calibration curve.

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

The concentrations from the reports are then incorporated with the purge and/or extraction data to arrive at a final concentration.

Water: Concentration (µg/L) = (C) (0.005L)/(V_s)

Soil: Concentration (mg/Kgdrywt) =
(C) ((((Vt+((M/100)(Vo)))(0.005L)/(Vo)(Vp)(100/100-M)(1/1000)

Where: C = Concentration calculated by Target in µg/L.
V_s = Volume of sample purged in L.
V_o = Weight of sample extracted in Kg.
V_t = Volume of methanol in L.
V_p = Total volume of extract purged in L.
M = Moisture in %.

7.6.2 Required Adjustment of Range Concentration Data: In order to minimize the "double counting" of the same hydrocarbon compounds on both the FID and PID chromatograms, the collective concentrations of MTBE, benzene, toluene, ethylbenzene, and m-, p- and o-Xylene identified on the PID chromatogram must be subtracted from the collective C_5 through C_8 and C_9 through C_{12} Aliphatic Hydrocarbon concentration value determined using the FID chromatogram.

7.7 Sample Analysis

7.7.1 PID Chromatogram

7.7.1.1 If desired, determine the peak area count for the Target VPH Analytes.

7.7.1.2 Determine the peak area count for the surrogate 2,5-dibromotoluene.

7.7.1.3 Determine the total area count for all peaks eluting 0.1 minutes after the retention time (RT) for o-Xylene and 0.1 minutes before the retention time for naphthalene.

7.7.1.4 Using the equations contained in Section 7.6.1, calculate the concentrations of the surrogate standard 2,5-dibromotoluene, and C_9 through C_{10} Aromatic Hydrocarbons. Optionally, calculate the individual concentrations of the Target VPH Analytes.

7.7.2 FID Chromatogram

7.7.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for n-pentane and 0.1 minutes before the Rt for n-nonane. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.7.2.2 Determine the total area count for all peaks eluting 0.1 minutes before the Rt for n-nonane and 0.1 before the RT for naphthalene. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.7.2.3 Determine the peak area count for the surrogate standard 2,5-dibromotoluene.

7.7.2.4 Using the equations contained in Section 7.6.1, calculate the concentrations of C_5 through C_8 Aliphatic Hydrocarbons, C_9 through C_{12} Aliphatic Hydrocarbons, and the surrogate standard 2,5-dibromotoluene.

7.7.3 Data Manipulations

7.7.3.1 By definition, the collective concentrations of aliphatic and aromatic fractions of interest **exclude** the individual concentrations of VPH Target Analytes. Accordingly, a series of data manipulation steps are necessary to adjust the collective range concentrations calculated in 7.7.1.4 and 7.7.2.4, to eliminate "double counting" of analytes.

7.7.3.2 Subtract the collective concentration of C_9-C_{10} Aromatic Hydrocarbons from the collective concentration of C_9-C_{12} Aliphatic Hydrocarbons.

7.7.3.3 Subtract the individual concentrations of the VPH Target Analytes from the appropriate aliphatic range (i.e., C_5-C_8 or C_9-C_{12} Aliphatic Hydrocarbons) in which they elute. If the individual concentrations of Target Analytes have not been quantitated, report the values as Unadjusted C_5-C_8 Aliphatic Hydrocarbons and Unadjusted C_9-C_{12} Aliphatic Hydrocarbons, and indicate “Not Determined” for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons.

7.7.4.1 If baseline correction is used, instrument baseline must be established for every batch of samples, and after the analysis of samples that are suspected to be highly contaminated.

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is

performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS/LCSD, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery.
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.9.

7.8.2 Surrogate recovery

All recoveries must meet the method acceptance limits of 70-130%.

The sample is evaluated for recoveries of the surrogate. If the surrogate recovery is outside of the method acceptance limits on one channel but acceptable on the other channel, narrate. If the recovery is high and the sample results are less than the PQL, narrate. If the recovery is low and may be attributable to matrix interference, reanalyze to confirm a matrix effect and narrate. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the soil % moisture is >25% and the surrogate recovery is >10%, narrate. If the soil reanalysis is still low, re-extract.

7.8.3 Chromatography

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, if the sample contains a concentration of the aliphatic range C_5 through C_8 which was integrated "valley to valley" instead of a "baseline to baseline"), it will be done in Target

Review. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration," current revision.

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7.9 Reporting

7.9.1 After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS or QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional forms, such as LCS/LCSD, chronology or calibration are generated in KIMS and/or QuickForms. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.9.2 The VPH reporting form contains an attestation that indicates whether significant modifications were made to the VPH method, a clear affirmation on whether the QA/QC procedures and standards specified in the method were followed and achieved.

7.9.3 Significant modifications may include the use of alternative detectors, the use of other than a purge-and-trap sample preparation, the use of solvents other than those recommended, and the use of a different surrogate than recommended. Any modifications which were made along with any QA/QC deviations are mentioned in the case narrative.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 General Requirements and Recommendations

8.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document the quality of data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.2 A methanol trip blank or acidified laboratory reagent grade water blank should continually accompany each soil sample or water sample batch, respectively, over the course of sampling, storage, and analysis.

8.1.3 A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

8.1.4 At a minimum, for each analytical batch (up to 20 samples), an Initial Calibration or opening and closing Calibration Check Standard, Laboratory Method Blank, Laboratory Control Sample (LCS) and LCS duplicate (LCSD) must be run. A duplicate sample (if client requested) and Matrix Spike (MS) and/or MS duplicate (MSD) should be analyzed, at the discretion of the analyst, based upon the nature of the sample. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered. The blank and spiked samples should be carried through all stages of the sample preparation and measurement process.

8.1.5 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration) **[REQUIRED]**
(2) Laboratory Method Blank **[REQUIRED]**
(3) Laboratory Control Sample and Duplicate **[REQUIRED]**
(4) Samples
(5) Duplicate sample [if client requested]
(6) Matrix Spike/Duplicate [As appropriate]
(7) Mid-range Calibration Check Standard [consider after 10 samples, as appropriate]

8.1.6 At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, reanalyze the sample.

8.2 Minimum Instrument QC

8.2.1 The n-pentane (C_5) and MTBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively. This is achievable using the recommended trap and purge-and-trap procedures. Coelution of the m- and p- xylene isomers is permissible. Any surrogates used must be adequately resolved from individual compounds in the VPH Component Standard.

8.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Section 7.3)

8.2.3 Calibration factors must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of the calibration factors may be assumed if the %RSD over the working range of the curve is less than or equal to 25%. (See Section 7.4)

8.3 Initial and Periodic Method QC Demonstrations

The procedures specified in Section 8.3.1.1 through 8.3.1.3 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

8.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate laboratory reagent grade water and/or clean sand blanks spiked with each analyte of interest at approximately half of the highest calibration standard (100 µg/L water and 16.7-50 mg/Kg soil).

8.3.1.1 Add an appropriate aliquot of the stock or primary dilution standard solution(s) to each of the four replicate laboratory reagent grade water or clean sand blanks. Purge and analyze each replicate according to the procedures described in Section 7.1.

8.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

8.3.1.3 For each analyte, the mean accuracy, expressed as a percentage of the true value, must be between 70% and 130%, and the %RSD must be less than or equal to 25%. Higher recoveries are permissible for n-nonane.

8.4 Ongoing Method QC Demonstrations

8.4.1 Each sample, blank, and Laboratory Control Sample must be spiked with the surrogate spiking solution. Required surrogate recovery is 70% to 130%. Recoveries outside of this range must be noted and discussed in the data report form.

8.4.2 At a minimum, with every batch of 20 samples or less the laboratory must analyze the following:

8.4.2.1 Calibration Check Standard - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended after the analysis of the tenth sample. If the percent difference (%D) of any analyte within the calibration check standard varies from the predicted response by more than 25%, a new calibration curve must be prepared for that analyte.

8.4.2.2 Laboratory Method Blank - A water or soil Laboratory Method Blank is prepared and analyzed. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest.

8.4.2.3 Laboratory Control Sample and Duplicate- The Laboratory Control Sample and Duplicate are prepared and analyzed. The spike recovery must be between 70% and 130%. RPD is <25%.

8.4.2.4 Sample duplicate - Sample duplicates may be laboratory duplicates or field duplicates per the client's request. The %RPD of duplicate samples must not exceed 50%.

8.4.2.5 System Solvent Blank - If baseline correction will be employed, as specified in Section 7.7.4, a system solvent blank, air blank, and/or system run must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated.

8.4.3 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) is performed per the client's request.

Matrix Spike (MS)/MS Duplicate (MSD) –The water or soil MS/MSD spike is prepared and analyzed.

The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. The %RPD of the MS/MSD should be less than or equal to 25%.

8.4.4 If any of the performance standards specified in Section 8.4.1 and 8.4.2 are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are fallen out must be rerun. If this is not possible, that data must be reported as suspect.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," for procedures on determining the MDL.

Refer to the current revision Method MADEP VPH-04-1.1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 8000B, 8015C, 5030B and the current edition of 8020.

Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), MADEP, May 2004, Revision 1.1.

Katahdin SOP CA-101, "Equipment Maintenance and Troubleshooting," current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," current revision.

LIST OF TABLES AND FIGURES

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP-VPH-04-1.1

QC check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point external calibration of 15 VPH component standards, and a surrogate. Also, collective calibrations of C_9 through C_{10} aromatic hydrocarbons, C_5 through C_8 aliphatic hydrocarbons and C_9 through C_{12} aliphatic hydrocarbons.	Initial calibration prior to sample analysis	The %RSD must be ≤ 25% See Sect. 7.4.1 and 7.4.2 for further information.	Investigate and repeat initial calibration
Initial Calibration Verification (ICV)	Once after each calibration	All analytes ≤ 25 %D of the expected value.	Reanalyze sample Reprepare standard Reprepare standard from fresh stock.
CV	If initial calibration analyzed, daily and after 20 samples, and at end of sequence.	%D for all analytes within ±25% (n-nonane ±30%)	Evaluate the samples: If the %D >±25% and sample results are < PQL, narrate. If %D >±25% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report samples results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One LCS/LCSD per prep batch	Spike recovery must be between 70% and 130%	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate	Every sample, blank, and QC sample	Recovery must be between 70% and 130%	Refer to section 7.8.2

TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP-VPH-04-1.1

QC check	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate	Per client request	Recovery must be between 70% and 130%.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample duplicate if requested by the client	One per batch of 20 samples	%RPD of duplicate must be less than 50%.	(1)check calculations for errors (2) Evaluate QC
Demonstration of ability to generate acceptance accuracy and precision using four replicate analyses of a QC check sample	Once per analyst initially and yearly thereafter	For each analyte, the mean accuracy must be between 70% and 130% and the %RSD must be $\leq$ 25%	Investigate; reprep
MDL and/or LOD/LOQ Verification	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications,” current revision.		

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-312-11	METHOD MADEP-VPH-04-1.1
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
Procedures	7.5.9 For non-target analytes and target analytes eluting in the aliphatic or aromatic fractions, the upper linear range of the system is based on the highest calibration standard. Refer to Table 3 for concentrations.	See Section 9.5.8
Procedures	7.5.4 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.	Section 9.5.4 Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 9.3.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 3

VPH CALIBRATION CONCENTRATIONS

Component	Level 1 µg/L	Level 2 µg/L	Level 3 µg/L	Level 4 µg/L	Level 5 µg/L	Level 6 µg/L
n-Pentane	1	5	10	50	100	300
2-Methylpentane	1	5	10	50	100	300
MTBE	1	5	10	50	100	300
2,2,4-Trimethylpentane	1	5	10	50	100	300
Benzene	1	5	10	50	100	300
Toluene	1	5	10	50	100	300
n-Nonane	1	5	10	50	100	300
n-Decane	1	5	10	50	100	300
n-Butylcyclohexane	1	5	10	50	100	300
Ethylbenzene	1	5	10	50	100	300
m,p-Xylene	2	10	20	100	200	600
o-Xylene	1	5	10	50	100	300
1,2,4-Trimethylbenzene	1	5	10	50	100	300
Naphthalene	1	5	10	50	100	300
2,5-Dibromotoluene	1	5	10	50	100	300
C_5-C_8	3	15	30	150	300	900
C_9-C_{12}	2	10	20	100	200	600
C_9-C_{10}	1	5	10	50	100	300

Amount of standard added to 5 mL volume: Tekmar System:

Level 1 1 µL of 5 µg/mL standard
Level 2 5 µL of 5 µg/mL standard
Level 3 10 µL of 5 µg/mL standard
Level 4 5 µL of 50 µg/mL standard
Level 5 10 µL of 50 µg/mL standard
Level 6 6 µL of 250 µg/mL standard

Amount of standard added to 50 mL volume: OI Analytical System:

Level 1 10 µL of 5 µg/mL standard
Level 2 50 µL of 5 µg/mL standard
Level 3 100 µL of 5 µg/mL standard
Level 4 50 µL of 50 µg/mL standard
Level 5 100 µL of 50 µg/mL standard
Level 6 60 µL of 250 µg/mL standard

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 4

HOLDING TIMES AND PRESERVATIVES FOR VPH SAMPLES

Matrix	Container	Preservation	Holding time
Aqueous Samples	40 mL VOC vials w/ Teflon-lined septa screw caps.	Add 3 to 4 drops of 1:1 HCl; cool to 4 (±2) °C.	14 days
Soil/Sediment Samples	40 mL VOC vials w/ Teflon-lined septa screw caps: add 15 g soil	1 mL methanol for every g soil; add before or at time of sampling; cool to 4 (±2) °C	28 days

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 5

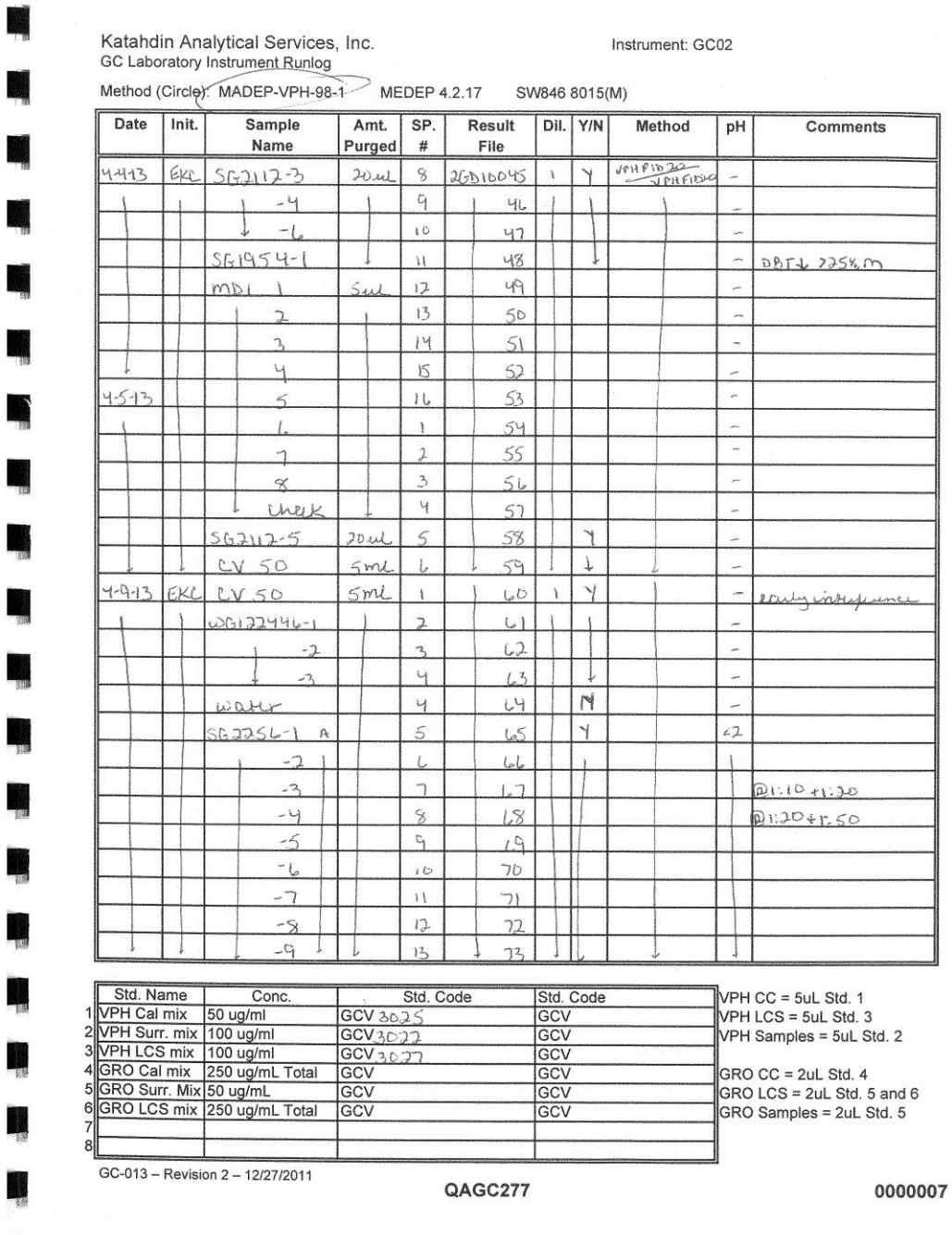
PQLS FOR METHOD MADEP VPH

Parameter/ Method	Analytes	Practical Quantitation Level	
		PQL (µg/L)	PQL (mg/Kg)
VPH/MADEP-VPH-04-1.1	Methyl-tert-butylether	5	0.25
	Benzene	3	0.25
	Toluene	5	0.25
	Ethylbenzene	5	0.25
	m- & p-Xylene	10	0.50
	o-Xylene	5	0.25
	Naphthalene	5	1.25
	C_5 - C_8 Aliphatics(FID)	100	25
	C_9 - C_{12} Aliphatics(FID)	100	25
	C_9 - C_{10} Aromatics(PID)	100	25

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



Katahdin Analytical Services, Inc. Instrument: GC02
GC Laboratory Instrument Runlog
Method (Circle): MADEP-VPH-98-1 MEDEP 4.2.17 SW846 8015(M)

Date	Init.	Sample Name	Amt. Purged	SP. #	Result File	Dil.	Y/N	Method	pH	Comments
4-4-13	EKL	SG2112-3	20mL	8	2GD10045	1	Y	VPH FID 2A / VPH FID 2B	-	
		-4		9	46				-	
		-6		10	47				-	
		SG1954-1		11	48				-	DBFM 225% rm
		MDL 1	5mL	12	49				-	
		2		13	50				-	
		3		14	51				-	
		4		15	52				-	
4-5-13		5		16	53				-	
		6		1	54				-	
		7		2	55				-	
		8		3	56				-	
		check		4	57				-	
		SG2112-5	20mL	5	58		Y		-	
		CV 50	5mL	6	59				-	
4-9-13	EKL	CV 50	5mL	1	60	1	Y		-	early instrument
		WG122446-1		2	61				-	
		-2		3	62				-	
		-3		4	63				-	
		WORK		4	64		N		-	
		SG2256-1 A		5	65		Y		42	
		-2		6	66					
		-3		7	67					@1:10 + 1:20
		-4		8	68					@1:20 + 1:50
		-5		9	69					
		-6		10	70					
		-7		11	71					
		-8		12	72					
		-9		13	73					

	Std. Name	Conc.	Std. Code	Std. Code
1	VPH Cal mix	50 ug/ml	GCV 3025	GCV
2	VPH Surr. mix	100 ug/ml	GCV3027	GCV
3	VPH LCS mix	100 ug/ml	GCV3027	GCV
4	GRO Cal mix	250 ug/mL Total	GCV	GCV
5	GRO Surr. Mix	50 ug/mL	GCV	GCV
6	GRO LCS mix	250 ug/mL Total	GCV	GCV
7				
8				

VPH CC = 5uL Std. 1
VPH LCS = 5uL Std. 3
VPH Samples = 5uL Std. 2

GRO CC = 2uL Std. 4
GRO LCS = 2uL Std. 5 and 6
GRO Samples = 2uL Std. 5

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TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Int.* ____ .) **DueDate** _____ .

Client:	**Verbal Review**	**Primary Review**	**Secondary Reveiw**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT ND's to* - *PQL* ☐ *MDL* ☐ *LOD* ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitaion Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐) .	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement _Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD O LAB O	☐ FORM 6 (s) DOD O LAB O
☐ FORM 4 (s)	☐ FORM 7 (s) DOD O LAB O
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ O MDL O LOD O
☐ FORM 8 (s)	☐ Flagging **B** O **L** O **M** O **C** O
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ O MDL O LOD O	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD O LAB O	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD O LAB O	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	Exceedences
< 11	0
11 to 30	1
31 to 50	2
51 to 70	3
71 to 90	4
> 90	5

QA-044 – Revision10 – 04/07/2017

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: ANTHONY BULLENTINI

Review Date: 021819

SOP Number: CA-312-11

SOP Title: METHOD FOR DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (MADEP-VPH)

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 2-20-19

QAO Signature: _____ **Date:** 02.21.19

KATAHDIN ANALYTICAL SERVICES,
STANDARD OPERATING PROCEDURE

SOP Number: CA-322
Revision History
Cover Page
Page 1

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS (MADEP - EPH)

Prepared By: Peter Lemay Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 2/12/01

Operations Manager: John C. Burton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2·12·01

General Manager: [signature] Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, other updates to sections 7, 8 and QA Table.	DN	2·12·01	2/12/01
02	Changes to sections 2.2, 5.2.2, 7.3.1.3, 7.5.1.1, 7.6.3.4, and 8.0. Also changes to tables 2, 3, and 4	DN	5·2·02	5·2·02
03	Added definitions and information for new data processing system. Added or changed wording to clarify section 7 and Table 2. Added wording to sections 8 + 9 per recent NELAC + Navy audit responses. Minor changes throughout. New figures	MRC	11.15.04	11.15.04
04	Changed terminology for LCS's: MS's. Corrected Soil H.T. Added required LCSD and breakthrough check. Updated reference. Included KIMS forms. Minor changes throughout.	LAD	051305	051305
05	Changed references from MADEP EPH-98-1 to MADEP EPH-04-1 fixed typographical and grammatical errors	LAD	04/06	04/06

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS (MADEP - EPH)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Many changes made throughout, including but not limited to, adding linear regression calibration, spike concentrations, %0 RSD criteria, PQL's, standard solvent, freezing of soil samples. Refer to QAM/SOP change form filed w/ SOP in QA for more details.	LAD	09/07	09/07
07	Sect. 2.1: Changed solvent from hexane to methylene chloride and added solvent exchange step.	LAD	03/08	03/08
08	Changed surrogate from 1-chloro-octadecane to alpha androstane. Added HPLC fractionation references. Updated MDL/LOD/LOQ verification criteria. Updated references. Updated Logbook page and Data Review Checklist.	LAD	04/10	04/10
09	Added ICV criteria. Removed RSD. Added % Difference. LAD 06.07.10 Added ~~Average~~ Average Calibration Model to sections 7.3, 7.5 and Table 1.	LAD	06/10	06/10
10	2.1-Samples extracted in MeCl2. Section 4.3.2-updated columns. Section 8.2.5- changed corrective action for failing C28-C20 ratio to maintenance. Table 1- Added fractionation check frequency to once per week for HPLC. Changed aliphatic standard solvent to hexane. Added HPLC overlay example. Updated Figures 1-3.	LAD	01/12	01/12
11	Sect. 5 & 8- Changed surrogate COD to o-terphenyl Sect. 4- Updated column info. Sect. 7- Updated program. Updated Figures 1→3.	LAD	04/13	04/13
12	Minor edits to clarify ICV frequency, report form generation and LCS/LCSD acceptance criteria. Added DoD QSM 5.0 reference. Updated review checklist. Changed KAS INC. to KAS throughout.	LAD	08/15	08/15
13	Sect. 1- Updated ending marker definition. Sect. 5- Added standards to title, corrected Table reference, corrected surr. conc., added HPLC, removed cartridge, corrected All. range. Sect. 7- Removed thermal bleed added solvent blank for baseline correction. Several edits for clarification. Sect. 8- removed excluding C36 for LCS recovery	LAD	04/16	04/16

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS (MADEP - EPH)

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 10- updated method references. Changed throughout - thermal bleed to solvent blank according to the method requirements.	LAD	09/17	09/17
15	Sect. 7. Added PAH Confirmation by GCMS SIM.	LAD	10/18	10/18

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-322-15**, titled **METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-322-15**, titled **METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD.**

Recipient: _____ Date: _____

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD

1.0 SCOPE AND APPLICATION

This method is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C_9 through C_{18}, and C_{19} through C_{36}. Extractable aromatic hydrocarbons are collectively quantitated within the C_{11} through C_{22} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 265 °C.

This method is based on a solvent extraction, HPLC or silica gel cartridge fractionation process, and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

This method is also able to measure the individual concentrations of Target Polynuclear Aromatic Hydrocarbons (PAH) Analytes, including Diesel PAH Analytes, in water and soil.

The fractionation step described in this method may be eliminated to allow for the determination of a Total Petroleum Hydrocarbon (TPH) value, and/or obtain qualitative “fingerprinting” information. While TPH provides little information on the chemistry, toxicity, or environmental fate of petroleum mixtures, it may be a cost-effective screening tool in cases where a relatively low concentration of contamination is suspected.

Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, or other petroleum products that contain a significant percentage of hydrocarbons lighter than C_9. This method, in and of itself, is also not suitable for the evaluation of petroleum products that contain a significant percentage of hydrocarbons heavier than C_{36}.

Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target PAH Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target or Diesel PAH Analyte. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analysis by dissimilar columns, gas chromatography/mass spectrometry (GC/MS) analysis, or other suitable technique is recommended in cases where a target PAH analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where coelution of a non-targeted hydrocarbon compound is suspected.

This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an

equivalent or superior level of performance. MADEP encourages methodological innovations which (a) better achieve method and/or data quality objectives, (b) increase analytical precision and accuracy, (c) reduce analytical uncertainties and expenses, and/or (d) reduce the use of toxic solvents and generation of hazardous wastes. Laboratories that modify this method must achieve all required performance and acceptance standards, and must have on file a Standard Operating Procedure which thoroughly describes the revised or alternative method, and documentation which demonstrates an equivalent or superior level of performance. All significant modifications to the method must be disclosed and described on the data report form.

1.1 Definitions

Aliphatic Hydrocarbon Standard is defined as a 14 component mixture of the normal alkanes listed in Table 3. The compounds comprising the Aliphatic Hydrocarbon Standard are used to (a) define and establish windows for the two aliphatic hydrocarbons ranges, and (b) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.

Analytical Batch is defined as a group of field samples with similar matrices that are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

Aromatic Hydrocarbon Standard is defined as a 17 component mixture of the polynuclear aromatic hydrocarbons (PAHs) listed in Table 3. The compounds comprising the Aromatic Hydrocarbon Standard are used to (a) define the individual retention times and chromatographic response factors for each of the PAH analytes listed in Table 3, (b) define and establish the window for the C_{11} through C_{22} Aromatic Hydrocarbon range, and (c) determine an average chromatographic response factor that can in turn be used to calculate the collective concentration

C_9 through C_{18} Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds eluting from n-nonane (n-C_9) to just before n-nonadecane (n-C_{19}).

C_{19} through C_{36} Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds eluting from n-nonadecane (n-C_{19}) just after n-hexatriacontane (n-C_{36}).

C_{11} through C_{22} Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds eluting from naphthalene just after Benzo(g,h,i)Perylene, excluding Target PAH Analytes.

Calibration Check Standard is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock standard solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

Calibration Standards are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

Diesel PAH Analytes are defined as naphthalene, 2-methylnaphthalene, phenanthrene, and acenaphthene, and are a subset of Target PAH Analytes. For most sites contaminated by a release of (only) diesel or #2 fuel oil, Diesel PAH Analytes will be the only Target PAH Analytes of interest.

Extractable Petroleum Hydrocarbons (EPH) are defined as collective fractions of hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. EPH is comprised of C_9 through C_{18} Aliphatic Hydrocarbons, C_{19} through C_{36} Aliphatic Hydrocarbons, and C_{11} through C_{22} Aromatic Hydrocarbons.

Field Duplicates are defined as two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

Fractionation Surrogate Standards are compounds that are spiked into the sample extract immediately prior to fractionation, in order to determine if significant quantities of naphthalene or substituted naphthalenes are being stripped into the aliphatic extract and to evaluate fractionation efficiency.

Instrument Solvent Blank is defined as an injection of solvent. The purpose of the Solvent Blank is to determine the level of noise and baseline rise attributable to the solvent, in the absence of any other analytes.

Laboratory Control Sample (LCS) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution. The LCS is prepared and analyzed in the same manner as the samples and its purpose is to determine the bias of the analytical method.

Laboratory Control Sample Duplicate (LCSD) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution, processed and analyzed in the same manner as the LCS. The analysis of the LCSD

gives a measure of the precision associated with laboratory procedures, but not with sample collection, preservation or storage procedures.

Matrix Duplicates (DUP) are defined as duplicate samples prepared and analyzed separately with identical procedures. For soils samples DUPs are taken from the same sampling container; for aqueous samples, a second sample container is used. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

Matrix Spike Sample (MS) is defined as an environmental sample that has been spiked with a matrix spiking solution containing known concentrations of method analytes. The MS sample is treated and analyzed exactly as other samples, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of an unspiked sample aliquot. The measured values in the MS sample must be corrected for background concentrations when calculating recoveries of spiked analytes.

Laboratory Method Blank is defined as an aliquot of laboratory reagent grade water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

Matrix Spiking Solution is defined as a solution prepared independently from the calibration standards, containing known concentrations of method analytes.

Surrogate Standards are compounds spiked into all samples, blanks, and matrix spikes to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

Target PAH Analytes are defined as the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 3.

Total Petroleum Hydrocarbons (TPH) are defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. TPH is equivalent to the summation of C_9 through C_{18} Aliphatic Hydrocarbons, C_{19} through C_{36} Aliphatic Hydrocarbons, and C_{11} through C_{22} Aromatic Hydrocarbons.

Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds eluting from naphthalene through benzo(g,h,i)Perylene.

Unadjusted TPH is defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, including the target PAH analytes.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect, chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of EPH by MADEP EPH-04-1. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of EPH by MADEP EPH-04-1 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS (material safety data sheet) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. EPH sample vials are considered “P” waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Please refer to the

current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, and concentrated in a Kuderna-Danish apparatus during which the extract is solvent exchanged into hexane. Sample separation into aliphatic and aromatic fractions is conducted using HPLC fractionation. The two extracts produced are then re-concentrated to final volumes of 1mL each (i.e., an aliphatic extract and an aromatic extract). The extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively integrated within the C_9 through C_{18} and C_{19} through C_{36} ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C_{11} through C_{22} range, and is (optionally) used to identify and quantitate individual concentrations of Target PAH Analytes.

2.2 Average calibration factors, determined using an aliphatic hydrocarbon standard mixture, are used to calculate the collective concentrations of C_9 through C_{18} and C_{19} through C_{36} aliphatic hydrocarbons. An average calibration factor determined using a PAH standard mixture is used to calculate a collective C_{11} through C_{22} aromatic hydrocarbon concentration. Calibration factors determined for individual components of the PAH standard mixture are also used to calculate individual concentrations of Target PAH Analytes.

2.3 This method is suitable for the analysis of waters, soils, and sediments.

2.4 This method is based on (1) USEPA Methods 8000, 8100, and 3630, SW-846, "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, III, IIIA, and IIIB 1996, 1998 & 2004,; (2) Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990; (3) "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992; and (4) Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004, revision 1.1.

3.0 INTERFERENCES

3.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride.

3.2 High purity reagents must be used to minimize interference problems.

3.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a system solvent blank to check for cross-contamination.

3.4 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled.

3.5 Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable and Total Petroleum Hydrocarbons.

3.6 Because of their weakly polar nature, naphthalene and substituted naphthalenes are readily fractionated into the aliphatic extract. By using HPLC fractionation this contamination is eliminated. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the evaluation of the aliphatic fraction for the presence of naphthalene and 2-methylnaphthalene in the LCS/LCSD pair on a batch basis. The fractionation surrogate, 2-Bromonaphthalene, is used to monitor sample-specific fractionation efficiency.

4.0 APPARATUS AND MATERIALS

4.1 The following glassware is used for this method:

4.1.1 auto sampler: 2mL glass vials with Teflon-lined rubber crimp caps

4.1.2 10mL vials with Teflon-lined caps

4.1.3 Class “A” volumetric flasks: 10, 25, 50 and 100mL

4.1.4 Class “A” volumetric pipettes: 1, 5 or 10mL

4.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1g must be used for weighing soil samples.

4.3 Gas Chromatograph

4.3.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph for use with capillary columns is required. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. The current system is a Hewlett Packard 5890 GC connected to the Turbochrom data acquisition system, which is connected to the Target data processing system.

4.3.2 Current columns are: Primary aromatic analysis – RTX-5: 30M x 0.53mm id x 0.5µm film thickness. Secondary aromatic or aliphatic analysis – RTX-1: 30M x 0.53mm id x 0.5µm film thickness

4.3.3 Detector: A Flame Ionization Detector (FID) is required.

4.3.4 Auto sampler: An auto sampler capable of making 1 to 4 µL injections is recommended.

4.4 Microsyringes: 10-µL, 100-µL, 250-µL, 500-µL, 1000-µL

5.0 REAGENTS AND STANDARDS

5.1 Reagents - Solvents: hexane, methylene chloride, and acetone; pesticide grade or better. Store reagents away from other solvents.

5.2 Stock Standard Solutions

Stock standard solutions at approximately 1000ng/µL are purchased as certified solutions.

5.2.1 Aromatic Hydrocarbon Standard: The Aromatic Hydrocarbon Standard consists of the 17 PAH compounds listed in Table 4, a surrogate compound, and fractionation surrogate compounds in methylene chloride.

5.2.2 Aliphatic Hydrocarbon Standard: The Aliphatic Hydrocarbon Standard consists of the 14 normal alkanes listed in Table 4, naphthalene, 2-methylnaphthalene, and a surrogate compound in hexane.

5.2.3 Petroleum Reference Standard: The Petroleum Reference Standard consists of an API or commercial diesel fuel. Prepare stock standard solutions by accurately weighing approximately 0.0100g of neat product. Dissolve neat product in hexane and dilute to volume in a 10mL volumetric flask.

5.3 Surrogate Standards

5.3.1 Surrogate standards are used to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

5.3.2 The recommended surrogate standards are 5-alpha androstane and ortho-terphenyl (OTP) which are available from Restek or similar supplier.

5.3.3 The surrogate standard 5-alpha androstane is purchased as a certified solution in hexane. This solution is added to the Aliphatic Hydrocarbon standard.

5.3.4 The surrogate standard OTP is purchased as a certified solution in methylene chloride. This solution is added to the Aromatic Hydrocarbon standard.

5.3.5 Surrogate Spiking Solution: The recommended surrogate spiking solution is comprised of a mixture of the o-terphenyl and 5-alpha androstane surrogate standards. Prepare a surrogate spiking solution that contains the surrogate standards at a concentration of 45 ng/µL in acetone. Each sample, blank, and matrix spike is fortified with 1.0mL of the surrogate spiking solution.

5.4 Fractionation Surrogate Standards

5.4.1 The fractionation surrogate standards are added to the sample (hexane) extract just prior to fractionation. The purpose of the fractionation surrogate standards is to monitor the efficacy of the fractionation process, and ensure that unacceptable quantities of naphthalene and substituted naphthalenes are not being stripped into the aliphatic extract.

5.4.2 The recommended fractionation surrogate standards are 2-Fluorobiphenyl and 2-Bromonaphthalene. Alternative fractionation surrogate compounds are permissible, provided that a demonstration is made that such compounds exhibit polarities/fractionation properties similar to naphthalene.

5.4.3 The fractionation surrogate standards are purchased as certified in Methylene Chloride. This solution is added to the Aromatic Hydrocarbon standard.

5.4.4 Fractionation Surrogate Spiking Solution: The recommended fractionation surrogate spiking solution is comprised of 2-Fluorobiphenyl and 2-Bromonaphthalene prepared in hexane at concentrations of 45 ng/µL. An

aliquot of 1mL of the fractionation surrogate spiking solution is added to the 1mL EPH sample extract prior to HPLC silica gel fractionation.

5.5 Matrix Spike Standard

5.5.1 Analytes from each hydrocarbon group (i.e., aromatic and aliphatic hydrocarbons) are used in a matrix spiking solution, which is prepared independently from the calibration standards.

5.5.2 The recommended spiking solution, consisting of all 14 normal alkanes and 17 PAHs as listed in Table 3, is prepared in acetone at concentrations of 90ng/µL each. Each selected matrix spike as well as laboratory control/laboratory control duplicate sample is fortified with 1.0mL of the matrix spike standard.

5.6 Fractionation Check Solution

5.6.1 The Fractionation Check Solution is used to monitor the fractionation efficiency of the HPLC silica gel column, and establish the optimum volume of hexane needed to sufficiently elute aliphatic hydrocarbons, but not strip aromatic hydrocarbons.

5.6.2 Prepare a Fractionation Check Solution in hexane containing 200ng/µL of the Aliphatic Hydrocarbon standard (C_9-C_{36} alkanes), Aromatic Hydrocarbon standard (targeted PAH analytes), and extraction surrogates o-terphenyl and 5-alpha androstane. The final solution will contain 14 alkanes, 17 PAHs, and extraction surrogates at concentrations of 200ng/µL each. Alternative concentrations are permissible.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps.

6.2 Soil and sediment samples are collected in wide-mouth glass jars with Teflon-lined screw caps.

6.3 Aqueous samples must be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5mL of 1:1HCl to a 1 liter sample. The use of alternative acids is permissible. Following collection and addition of acid, the sample must be cooled to 4°C (±2°C).

6.4 Soil and sediment samples must be cooled to 4°C (±2°C) immediately after collection.

6.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

6.6 Aqueous samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

6.7 Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

A summary of sample collection, preservation, and holding times is provided in Table 3.

Refer to Katahdin SOP CA-511, Extraction of Petroleum Hydrocarbons from Samples for Analysis by MADEP - EPH Methods, current revision.

7.0 PROCEDURES

7.1 GC Conditions

Refer to instrument logbook for the current column and conditions.

Typical conditions are:

- Oven Program:
  - Set oven temperature to 70°C
  - then 10°C/min to 180°C
  - then 7°C/min to 310°C and hold for 8.5 minutes.
  - Total run time is 58.80 minutes.
- Sample injection is 1µL.
- The carrier gas is helium.
- The carrier gas Flow: 6mL/min.
- Air: 400mL/min.
- Make up gas flow: 30mL/min.
- FID temperature, 310°C
- Injection port temperature, 300°C
- GC operated in split/splitless mode

7.1.2 GC Maintenance

7.1.2.1 Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert.

7.1.2.2 Break off the first few inches, up to one foot, of the injection port side of the column.

7.1.2.3 Remove the column and solvent backflush according to the manufacturer's instructions.

7.1.2.4 Bake out the column at 300°C. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.

7.2 Retention Time Windows

7.2.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of the Aromatic Hydrocarbon and Aliphatic Hydrocarbon standard mixtures throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.

7.2.2 Calculate the standard deviation of the three absolute retention times for each individual component in the Aromatic Hydrocarbon standard, the Aliphatic Hydrocarbon standard, and all surrogate, fractionation surrogates.

7.2.3 Plus or minus three times the standard deviation of the absolute retention times for each standard should be used to define the retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.2.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window or use 0.1 minutes as a default value.

7.2.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

7.2.6 EPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of the beginning marker compound and ending 0.1 minutes

after the RT of the ending marker compound, except for n-C_{19}, which is both a beginning and ending marker compound for two different ranges.
The C_9 - C_{18} Aliphatic Hydrocarbon range ends immediately (0.1 min) before the elution of the n-C_{19} peak. The C_{19} - C_{36} Aliphatic Hydrocarbon range begins 0.1 before the elution of the n-C_{19} peak; therefore there is no overlap of the two ranges and the n-C_{19} peak is only included in the C_{19} - C_{36} Aliphatic Hydrocarbon range.

EPH marker compounds and windows are summarized in the table below.

Table - EPH Marker Compounds

Range/ Hydrocarbon Standard	Beginning Marker Cpd.	Ending Marker Compound
C_9-C_{18} Aliphatic Hydrocarbons	n-Nonane	Just Before n-Nonadecane
C_{19}-C_{36} Aliphatic Hydrocarbons	n-Nonadecane	n-Hexatriacontane
C_{11}-C_{22} Aromatic Hydrocarbons	Naphthalene	Benzo (g,h,i) Perylene

7.2.7 If a TPH analysis is done without fractionation, TPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of n-Nonane and ending 0.1 minutes after the RT of n-Hexatriacontane.

7.3 Calibration

Average calibration factors or linear regression is used to calculate the slope and y-intercept that best describes the linear relationship between EPH target analyte and range concentrations and instrument response.

Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standard solutions to volumetric flasks and diluting to volume with methylene chloride for the Aromatic standards and hexane for the Aliphatic standards. The surrogate OTP and the fractionation surrogate standards are added to the Aromatic Hydrocarbon Standard; the surrogate 5-alpha androstane is added to the Aliphatic Hydrocarbon Standard. One of the calibration standards must be at the concentration of the Reporting Limit. The other concentrations must correspond to the expected range of concentrations found in real samples or should define the working range of the detector. The following calibration levels are recommended: 1, 10, 50, 100, and 200ng/µL for the individual components. The individual and collective concentrations of standard analytes within each hydrocarbon range for these recommended calibration levels are provided in the table below.

Table - Recommended Calibration Standard Concentrations and masses (1µL Injection)

Component	Conc. of std. analytes (µg/ml)				
	1	10	50	100	200
Total Mass C_9 - C_{18} Aliphatic Hydrocarbons, ng (6 components)	6	60	300	600	1200
Total Mass C_{19} - C_{36} Aliphatic Hydrocarbons, ng (8 components)	8	80	400	800	1600
Total Mass C_{11}-C_{22} Aromatic Hydrocarbons/PAHs, ng (17components)	17	170	850	1700	3400

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak area for each compound.

7.3.1. Linear Regression

A calibration curve is prepared using area responses versus concentration. A linear calibration applying a first order equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient (r) must be greater than or equal to 0.990. The equation is:

$y = mx + b$

where: y = Instrument response
m = Slope of the line
x = Concentration of the calibration standard or range
b = The intercept

Calculate a linear regression (LR) for the individual PAH compounds that comprise the Aromatic Hydrocarbon standard. This is not necessary if the Target or Diesel PAH Analytes will not be individually identified and quantitated using the EPH method.

Calculate a LR for the extraction and fractionation surrogates.

A collective calibration curve must also be established for each hydrocarbon range of interest using the FID chromatogram of the appropriate fraction: C_9-C_{18} Aliphatic Hydrocarbons, C_{19}-C_{36} Aliphatic Hydrocarbons, and C_{11}-C_{22} Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. A listing of the collective concentrations of standards within each hydrocarbon range is provided in the table in section 7.3.

Note: The area for the surrogates must be subtracted from the area summation of the range in which they elute (e.g., 5-alpha androstane is subtracted from the C_{19} - C_{36} Aliphatic Hydrocarbon range). Do not include the area of naphthalene or 2-methylnaphthalene in the linear regression analysis of the C_9-C_{18} Aliphatic Hydrocarbon range.

7.3.2 The average calibration factor procedure

The ratio of area response to the concentration injected, defined as the calibration factor (CF), may be calculated for target PAH compounds using the equation below.

Calibration Factor (CF) = area of peak/concentration injected (ug/L)

The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest as determined using the equation below. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.

%RSD = (stand dev of 5 CFs/mean of 5 CFs) x 100

A collective calibration factor must also be established for each hydrocarbon range of interest using the FID chromatogram of the appropriate fraction: C_9-C_{18} Aliphatic Hydrocarbons, C_{19}-C_{36} Aliphatic Hydrocarbons, and C_{11}-C_{22} Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The results can be used to calculate the ratio of the peak area response summation to the concentration injected, defined as the CF, for the hydrocarbon ranges using the equation below. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest. A listing of the collective concentrations of standards within each hydrocarbon range is provided in the table in section 7.3.

$$\text{Range CF} = \frac{\text{Area Summation of Range Components}}{\text{Total concentration injected}(\text{ng}/\mu\text{l})}$$

Note: The area for the surrogates must be subtracted from the area summation of the range in which they elute (e.g., 5-alpha androstane is subtracted from the C_{19} - C_{36} Aliphatic Hydrocarbon range). Do not include the area of naphthalene or 2-methylnaphthalene in the analysis of the C_9-C_{18} Aliphatic Hydrocarbon range.

7.3.3. For TPH analyses, without fractionation, calculate a collective calibration curve. Tabulate the summation of the peak areas of all component standards in the aliphatic fraction (i.e., 14 components) against the total mass injected. Do not include any surrogates.

7.3.4 At a minimum, the working calibration curve must be verified on each working day, after every 20 samples or 24 hours (whichever is more frequent), and at the end of the sequence, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than ± 25% (± 30% for n-nonane), as calculated using the equation below, instrument maintenance must be performed (such as changing the liner) and/or a new calibration curve must be determined for that analyte or range.

Linear Calibration Percent Drift (%D)

$$\%D = (R_1 - R_2 / R_2) \times 100$$

where: R1 = Calculated concentration from curve.
R2 = Expected concentration.

For the closing continuing calibration standard, four compounds may exhibit percent differences or percent drifts greater than 25 % but less than 40 %.

Average Calibration Factor Percent Difference (%D)

$$\%D = (CFavg - CFcc)/(CFavg) \times 100$$

where: CFavg = Average calibration factor calculated from initial calibration
CFcc = Calibration factor calculated from continuing calibration

7.4 GC Analysis

7.4.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration and a independent calibration verification standard (or calibration verification) followed by sample extracts interspersed with blanks and QC samples, and closes with a mid-range continuing calibration verification. The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.4.2 Aliphatic and aromatic extracts are introduced into the gas chromatograph by direct injection of 1µl of sample.

7.4.3 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.2. Alternately, the default value of 0.1 minutes may be used for the daily retention time window.

7.4.3.1 Tentative identification of an aromatic analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is performed by reanalysis on a dissimilar GC column or by GC/MS SIM. Please refer to Katahdin SOP CA-213, current revision for GC/MS SIM analysis.

7.4.3.2 Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

7.4.4 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peak eluted between specified range “marker” compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual check be initially performed, to document proper integration functions.

7.4.5 When quantifying on a peak area basis by internal or external calibration, collective peak area integration for the fractional ranges, or TPH, must be from baseline (i.e. must include the unresolved complex mixture "hump" areas). For the integration of individual Target Analytes, surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.

7.4.6 Baseline correction using an instrument solvent blank is permissible, if conducted in accordance with the procedures and requirements specified in Section 7.6.5.

7.4.7 If the concentration of a Target or Diesel PAH Analyte, the aliphatic range C_9 through C_{18}, aliphatic range C_{19} through C_{36}, or aromatic range C_{11} through C_{22} exceed(s) the calibration range of the curve, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks

are on scale, and bracketed by upper and lower calibration standards. Overlapping peaks are not always evident when peaks are off scale.

7.4.8 For non-target peaks eluting in the aliphatic, aromatic or TPH fractions, the upper linear range of the system should be defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range for the detector. If any non-target peak eluting within any aliphatic or aromatic range exceeds twice the peak height documented for the highest range-specific calibration standard, dilute the extract and reanalyze.

7.5 Calculations

7.5.1 Linear Regression Analysis

The concentration of each analyte and/or hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, using linear regression analysis.

7.5.1.1. The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

$$Amt = (y - b) / m$$

Where: Amt = Concentration calculated by Target in ug/mL.
y = Instrument response
m = Slope of the line
b = the intercept

7.5.2 Average Calibration Factor Analysis

The concentration of each analyte and/or hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, using CF's.

7.5.1.2. The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

Amt = x/m

Where: Amt = Concentration calculated by Target in ug/mL.
x = Instrument response
M = average CF

7.5.3 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = Amt x DF [(Vt / Vo) x 1000]

Soil: Concentration (mg/Kgdrywt) = Amt x DF [(Vt / Vo) x (100/(100-M))]

Where: DF = Dilution factor.
Vt = Final extract volume in L.
Vo = Sample volume in L or kg.
M = % Moisture.

7.6 Sample Analysis

7.6.1 Aliphatic Fraction

7.6.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C_9 and 0.1 minutes before the Rt for n-C_{19}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.1.2 Determine the total area count for all peaks eluting 0.1 minutes before the Rt for n-C_{19} and 0.1 minutes after the Rt for n-C_{36}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.1.3 Determine the peak area count for the sample surrogate standard (5-alpha androstane) and any internal standard used. Subtract these values from the collective area count value within the appropriate hydrocarbon range(s).

7.6.1.4 Using linear regression in Target, calculate the collective concentrations of C_9 through C_{18} Aliphatic Hydrocarbons, C_{19} through C_{36} Aliphatic Hydrocarbons, and individual concentrations of any sample surrogate.

7.6.2 Aromatic Fraction

7.6.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.

7.6.2.2 Determine the peak area count for the sample surrogate (OTP), the fractionation surrogates, and any internal standard used. Subtract these values from the collective area count.

7.6.2.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

7.6.2.4 Using linear regression in Target, calculate the concentrations of Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons, the sample surrogate standard (OTP), the fractionation surrogates, and, optionally, the Target or Diesel PAH Analytes.

7.6.2.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the concentration of Unadjusted C_{11} - C_{22} Aromatic Hydrocarbons. If the concentration of Target or Diesel PAH Analytes were not determined.

7.6.3 Total Petroleum Hydrocarbons

7.6.3.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for n-C_9 and 0.1 minutes after the RT for n-C_{36}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.3.2 Determine the peak area count for any surrogate. Subtract these values from the collective area count value.

7.6.3.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

7.6.3.4 Using linear regression in Target, calculate the concentration of Unadjusted TPH, and, optionally, the Target or Diesel PAH Analytes.

7.6.3.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the concentration of unadjusted TPH. If the concentration of Target or

Diesel PAH Analytes were not determined, report a value for Unadjusted TPH, and indicate "Not Determined" for TPH.

7.6.4 Data Manipulations

7.6.4.1 By definition, the collective concentration of the aromatic fraction (and/or TPH) **excludes** the individual concentrations of the Target PAH Analytes. Accordingly, a data manipulation step is performed in KIMS that subtracts the individual PAH analyte concentrations.

7.6.4.2 Subtract the individual concentrations of the Target or Diesel PAH Analytes from the collective concentration of Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons and/or Unadjusted TPH. If the individual concentrations of Target Analytes have not been quantitated, report a value for Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons and/or Unadjusted TPH, and indicate "Not Determined" for C_{11} through C_{22} Aromatic Hydrocarbons and/or TPH.

7.6.5 Baseline Correction for Instrument Noise Level

7.6.5.1 Range integration areas are corrected by the automatic subtraction of the baseline established by activation of a solvent blank.

7.6.5.2 The instrument baseline must be established by the direct injection of a system solvent blank. The injection of an air blank or activation of a temperature programmed chromatographic run without the injection of any material should be used to verify that the system noise is not attributable to solvent contamination. All system operational elements and parameters must be identical to those of a typical sample run.

7.6.5.3 If baseline correction is used, the baseline must be re-established for every analytical batch by the analysis of a System Solvent Blank. Baseline correction for EPH aliphatic and aromatic hydrocarbon area data may not be used for any sample for which the area count associated with the baseline correction is greater than 10% of the uncorrected area count for the sample's corresponding collective range.

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS/LCSD, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the method acceptance limits of 40-140%. The sample is evaluated for recoveries of the surrogates. For the aliphatic extract, if the recovery of 5-alpha androstane is high and the sample results are less than the PQL, narrate. If the recovery is low and may be attributable to matrix interference, reanalyze to confirm a matrix effect and narrate. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract.

For the aromatic fraction, if the recovery of OTP is low and the fractionation surrogates are low, refractionate. If the fractionation surrogates are acceptable and the OTP is low and may be attributable to matrix interference, reanalyze to confirm a matrix effect and narrate. If the OTP is low and is not attributable to matrix interference, the sample results are less than the PQL, and the fractionation surrogates are acceptable, re-extract.

7.7.3 Chromatography

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of

concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Any manual integrations that are necessary (for instance, if the sample contains a concentration of TPH/DRO which was integrated "valley to valley" instead of a "baseline to baseline"), are performed in Target Review. An "m" qualifier will automatically be printed on the quantitation report summary. The analyst must also date and initial in the space next to each of these qualifiers. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

7.7.4 Target Compound Detection

The aromatic analysis chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±50%, the concentration from channel A is reported.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the RL (Reporting Limit – based on the lowest calibration standard), if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ drastically, or if an analyte is present but it's retention time is outside of the retention time window for that analyte.

7.8 Reporting

7.8.1 After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms and KIMs. Depending on the QC level requested by the client, reports, such as chronology or calibration forms, are generated. Reports of Analysis (ROA), LCS/LCSD, MS/MSD and surrogate forms are generated in KIMS. The Analytical Sequence Form (Form 8) is generated in QuickForms. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is

provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8.2 The EPH reporting form contains an attestation, which indicates whether significant modifications were made to the EPH method, a clear affirmation on whether the QA/QC procedures and standards specified in the method were followed and achieved.

Significant modifications may include a different fractionation procedure than the one specified in the method, a change in the extraction procedure, the use of different surrogates. Any modifications, which were made along with any QA/QC deviations, are mentioned in the case narrative.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 4 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 4, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 4 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 4 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 General Requirements and Recommendations

8.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an

initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated.

8.1.2 A system solvent blank must be run after a sample suspected of being highly contaminated to determine if sample carryover has occurred.

8.1.3 At a minimum, for each analytical batch (up to 20 samples), a beginning and ending Calibration Check Standard, Method Blank, Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) must be run, and a Matrix Sample (MS) and/or MS duplicate or sample duplicate (DUP) should be analyzed, at the discretion of the analyst, based upon the nature of the sample. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered. The blank and spiked samples should be carried through all stages of the sample preparation and measurement process.

8.1.4 The recommended sequence of analysis is as follows:

- Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration) [REQUIRED]
- Method Blank [REQUIRED]
- Laboratory Control Sample and Laboratory Control Sample Duplicate [REQUIRED]
- Samples [up to 20]
- Matrix Sample/duplicate [As appropriate/Client Requested]
- Mid-range Calibration Check Standard [consider after 10 samples, as appropriate] [REQUIRED after 20 samples or at end of analytical sequence]

8.1.5 At a minimum, when the surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, reanalyze the sample.

8.1.6 Each sample and QC sample must be evaluated for potential breakthrough on a sample-specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic fractions of the LCS and LCSD. If the concentration of either naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration (sum of analyte in aromatic and aliphatic fraction) for

naphthalene and 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all archived batch extracts. If the fractionation surrogate recovery is outside the 40-140% limits, then fractionation must be repeated on the archived extract of the affected samples.

8.2 Minimum Instrument QC

8.2.1 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

8.2.1.1 The n-nonane (n-C_9) peak must be adequately resolved from the solvent front of the chromatographic run.

8.2.1.2 The surrogates O-TERPHENYL and 5-alpha androstane, fractionation surrogate standards must be adequately resolved from any individual components in the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.

8.2.1.3 All peaks of interest from the Aliphatic Hydrocarbon standard must be adequately resolved to baseline. In the Aromatic Hydrocarbon standard, baseline separation is expected for Phenanthrene and Anthracene. Benzo(a)Anthracene, Chrysene, Benzo(b)Fluoranthene, Benzo(k)flouranthene, Dibenzo(a,h)Anthracene, and Indeno(1,2,3-cd)Pyrene are not expected to be chromatographically separated to baseline; however, sufficient separation should be obtained to 50% baseline.

8.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.

8.2.3 Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels and the correlation coefficient (r) must be at least 0.990.

8.2.4 The calibration ranges of the aliphatic and aromatic hydrocarbon fractions, and/or TPH fraction, are based on the respective high concentration standard.

8.2.5 In order to demonstrate the absence of mass discrimination, the response ratio of C_{28} to C_{20} must be at least 0.85. If <0.85, instrument or injection port maintenance is necessary. The chromatograms of Continuing Calibration Standards for aromatics must also be reviewed to ensure that there are no obvious signs of mass discrimination.

8.2.6 Due care must be exercised to assure that the peaks for naphthalene and n-dodecane in the aliphatic hydrocarbon fraction are resolved to allow for an accurate determination of the naphthalene concentration in the LCS/LCSD pair.

8.3 Initial and Periodic Method QC Demonstrations

The procedures specified in Section 8.3.1 through 8.3.3 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

8.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate laboratory reagent grade water and/or clean sand blanks spiked with each analyte of interest at approximately 50µg/L and/or 5mg/kg, respectively.

8.3.1.1 Extract each replicate according to the procedures described in the current revision of Katahdin SOP CA-511. Analyze according to the procedures described in section 7.0.

8.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

8.3.1.3 For each analyte, excluding n-C_{36}, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%. Poorer recoveries may be experienced for the n-C_{36} standard. For each analyte, the %RSD must be less than or equal to 25% (30% for n-nonane).

8.3.2 Fractionation

8.3.2.1 To demonstrate the capability of properly fractionating aliphatic and aromatic hydrocarbons in a sample, the analyst must first prepare and analyze the Fractionation Check Solution specified in Section 5.6, using the HPLC fractionation procedure.

8.3.2.2 For each analyte within the Fractionation Check Solution, excluding n-C_{36}, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%.

8.4 Ongoing Method QC Demonstrations

8.4.1 Each sample, blank, and LCS/LCSD must be spiked with the surrogate spiking solution. Required surrogate recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

8.4.2 Each sample extract must be spiked with a fractionation surrogate spiking solution prior to fractionation. Required recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

8.4.3 At a minimum, with every batch of 20 samples or less the lab must analyze the following:

8.4.3.1 Calibration Check Standard - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to and after sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered after the analysis of the tenth sample. If the percent difference (%D) of any analyte, within a calibration check standard, varies from the predicted response by more than 25% (30% for n-nonane), instrument maintenance must be performed (such as changing the liner) and/or a new calibration curve must be prepared for that analyte.

8.4.3.2 Method Blank - A water or soil Method Blank is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the surrogate spiking solution. Peaks detected within the retention time window of any analyte or range of interest above a Reporting Limit must be noted on the data report form.

8.4.3.3 Laboratory Control Sample - A Laboratory Control Sample is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the matrix spiking solution. The spike recovery should be between 40% and 140%.

8.4.3.4 Laboratory Control Sample Duplicate - A Laboratory Control Sample duplicate is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the matrix spiking solution. The

spike recovery should be between 40% and 140%, excluding n-C_{36}. The RPD between the laboratory control sample and laboratory control sample duplicate should be less than 25%.

8.4.3.5 Solvent Blank - If baseline correction will be employed, a solvent blank must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated.

8.4.4 At the discretion of the analyst, and in consideration of sample matrices and data quality objectives, it is recommended that with every batch of 20 samples or less the lab consider analysis of the following:

8.4.4.1 Matrix Sample (MS)/Duplicate (MSD) - The water or soil MS is prepared by fortifying an actual water or soil sample with 1.0mL of the matrix spiking solution. The purpose of the MS spike is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations. The corrected concentrations of each analyte within the MS spike sample should be within 40 to 140% of the true value. The %RPD of MS Duplicates should be less than 50%.

8.4.5 If any of the performance standards specified in Section 8.4.1 through 8.4.3 are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are fallen out must be rerun. If this is not possible, that data must be reported as suspect.

8.4.6 The analyte and hydrocarbon range reporting limits should be verified/re-established at least once per year, or upon a major change in system equipment or operations.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

Limits of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be

laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the anaysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

The Practical Quantitation Limit (PQL) concentrations for all the target analytes are listed in Table 5.

Refer to the current revision Method MADEP EPH-04-1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004 revision 1.1

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015).

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

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Katahdin SOP CA-101, “Equipment Maintenance and Troubleshooting”, current revision.

Katahdin SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.

Katahdin SOP CA-511, “Extraction of Petroleum Hydrocarbons from Samples for Analysis by MADEP - EPH Methods”, current revision.

LIST OF TABLES AND FIGURES

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TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP EPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point external calibration of 17 targeted PAH standards, and collective calibrations of C_{11} through C_{22} aromatic hydrocarbons, C_9 through C_{18} aliphatic hydrocarbons and C_{19} through C_{36} aliphatic hydrocarbons.	Initial calibration prior to sample analysis	The correlation coefficient (r) must be greater than or equal to 0.99. CF must be equal to or less than 25%	Investigate and repeat initial calibration
Initial Calibration Verification (ICV)	Once after each calibration	All analytes $\leq$ 25 %D of the expected value.	Reanalyze sample Reprepare standard Reprepare standard from fresh stock.
CV	If initial calibration analyzed, daily and after 10 to 20 samples, and at end of sequence.	%RPD within 30% for n-nonane and for all other analytes within 25% The closing CCV may have up to 4 compounds > 25% but less than 40% D.	Evaluate the samples: If the %RPD >25% (30% for n-nonane) and sample results are < PQL, narrate. If %RPD >25% (30% for n-nonane) and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. if the blank results are above the PQL, report samples results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/LCSD	One LCS/LCSD pair per prep batch	Spike recovery must be between 40% and 140% and RPD less than 25%	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
LCS/LCSD	One LCS/LCSD pair per prep batch	Concentration of naphthalene and 2-methylnaphthalene in the aliphatic fraction must be less than 5% of total concentration	Refractionate all archived extracts from the extraction batch
Surrogate	Every sample, blank, and QC sample	Recovery must be between 40% and 140%	Refer to section 7.7.2

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TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP EPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate	One MS/MSD per batch of 20 samples (optional), as requested by clients.	Recovery must be between 40% and 140% and RPD less than 50%	(1) Evaluate the samples and associated QC: i.e. If the LCS is acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample duplicate if requested by the client	One per batch of 20 samples	%RPD of duplicate must be less than 50%.	(1)check calculations for errors (2) Evaluate QC
Fractionation Check Solution	HPLC - Once per week or sooner if suspect. Silica Gel Cartridges – once per lot or every 3 months, whichever is sooner	Mean accuracy must be between 40 and 140%.	Investigate and refractionate.
Demonstration of capability - seven replicate analyses of a QC check sample	One time per analyst initially and annually thereafter	For each analyte, the mean accuracy must be 40 to 140 %R and the %RSD must be $\leq$ 25%	Investigate; reprep
MDL and/or LOD/LOQ Verifications	Refer to the current revision of Katahdin SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, for procedures on determining the MDL.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-322-15	METHOD MADEP - EPH
Apparatus/ Materials		
Reagents/ Standards		
Sample preservation/ handling	1) Soil samples collected in clear jars	1) Soil samples collected in amber jars
Procedures	1) Prepare petroleum reference spiking solution by weighing 0.0100g of neat product	1) Prepare petroleum reference spiking solution by weighing 0.0250g of neat product
QC - Standards	1) Surrogate, spike and fractionation surrogate all prepared at 90 ug/mL for use in a 2 mL or prefractionated volume. 2) Fractionation check solution: aliphatics, aromatics, 5-alpha androstane, and o-terphenyl at 200ug/mL each	1) Surrogate at 40 ug/mL, matrix spike at 50-150 ug/mL, fractionation surrogate at 40 ug/mL. 2) Fractionation check solution: aliphatics and aromatics at 200ug/mL each
QC - LCS		
QC - Accuracy/ Precision		
QC - MDL		

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TABLE 3

HOLDING TIMES AND PRESERVATIVES FOR EPH SAMPLES

MATRIX	CONTAINER	PRESERVATION	HOLDING TIME
Aqueous Samples	1 Liter amber glass bottle with Teflon- lined screw cap	Add 5mL of 1:1HCl; cool to 4°C	Samples must be extracted within14 days and extracts analyzed within40 days of extraction
Soil/Sediment Samples	4-oz. (120mL) Wide-mouth glass jar with Teflon-lined screw cap	Cool to 4°C	Samples must be extracted within14 days and extracts analyzed within40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap. Jar should be filled to only 2/3 capacitiy to avoid breakage if expansion occurs during freezing.	Freeze at -10 °C in the field or in the laboratory*	Samples must be extracted within 14 days of the date thawed and extracts analyzed within 40 days of extraction*

*Samples processed in the laboratory must be preserved at 4 (±2) °C and frozen within 48 hours of the time of collection. Frozen samples may be held for up to one year prior to analysis and must be extracted within 24 hours of thawing.

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TABLE 4

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, MATRIX SPIKE AND CALIBRATION STANDARD CONCENTRATIONS - MADEP - EPH

	Stock *	Fractionation **	Matrix Spike	Calibration Standard Concentrations (in $MeCl_2$)				
ANALYTE	Standards (ng/µL)	Check Solution (ng/µL)	Standard *** (ng/µL)	(ng/µL) level 1	(ng/µL) level 2	(ng/µL) level 3	(ng/µL) level 4	(ng/µL) level 5
Naphthalene	1,000	200	90	1	10	50	100	200
2-Methylnaphthalene	1,000	200	90	1	10	50	100	200
Acenaphthylene	1,000	200	90	1	10	50	100	200
Acenaphthene	1,000	200	90	1	10	50	100	200
Fluorene	1,000	200	90	1	10	50	100	200
Phenanthrene	1,000	200	90	1	10	50	100	200
Anthracene	1,000	200	90	1	10	50	100	200
Fluoranthene	1,000	200	90	1	10	50	100	200
Pyrene	1,000	200	90	1	10	50	100	200
Benzo(a)Anthracene	1,000	200	90	1	10	50	100	200
Chrysene	1,000	200	90	1	10	50	100	200
Benzo(b)Fluoranthene	1,000	200	90	1	10	50	100	200
Benzo(k)Fluoranthene	1,000	200	90	1	10	50	100	200
Benzo(a)Pyrene	1,000	200	90	1	10	50	100	200
Indeno(1,2,3-cd)Pyrene	1,000	200	90	1	10	50	100	200
Dibenzo(a,h)Anthracene	1,000	200	90	1	10	50	100	200
Benzo(g,h,i)Perylene	1,000	200	90	1	10	50	100	200
Ortho-Terphenyl (surr)	1,000	200	----	1	10	50	100	200
Nonane	1,000	200	90	1	10	50	100	200
Decane	1,000	200	90	1	10	50	100	200
Dodecane	1,000	200	90	1	10	50	100	200
Tetradecane	1,000	200	90	1	10	50	100	200
Hexadecane	1,000	200	90	1	10	50	100	200
Octadecane	1,000	200	90	1	10	50	100	200
Nonadecane	1,000	200	90	1	10	50	100	200
Eicosane	1,000	200	90	1	10	50	100	200
Docosane	1,000	200	90	1	10	50	100	200
Tetracosane	1,000	200	90	1	10	50	100	200
Hexacosane	1,000	200	90	1	10	50	100	200
Octacosane	1,000	200	90	1	10	50	100	200
Triacontane	1,000	200	90	1	10	50	100	200
Hexatriacontane	1,000	200	90	1	10	50	100	200
5-alpha androstane(surrogate)	1,000	200	------	1	10	50	100	200

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TABLE 4, cont'd

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, MATRIX SPIKE AND CALIBRATION STANDARD CONCENTRATIONS - MADEP - EPH

* The Aromatic Hydrocarbon Standards (17 PAH compounds and ortho-Terphenyl) should be prepared in methylene chloride.
* The Aliphatic Hydrocarbon Standards (14 normal alkanes and 5-alpha androstane) should be prepared in hexane.
** The Fractionation Check Standard should be prepared in hexane.
*** The Matrix Spike Solution should be prepared in acetone.

TABLE 5

PQLs FOR METHOD MADEP EPH

PARAMETER/ METHOD	ANALYTE	PRACTICAL QUANTITATION LEVEL (PQL)	
		Aqueous (µg/L)	Soil (mg/kg)
Extractable Petroleum Hydrocarbons	Naphthalene	2	0.2
	2-Methylnaphthalene	2	0.2
	Acenaphthylene	2	0.2
	Acenaphthene	2	0.2
	Fluorene	2	0.2
	Phenanthrene	2	0.2
	Anthracene	2	0.2
	Fluoranthene	2	0.2
	Pyrene	2	0.2
	Benzo(a)Anthracene	2	0.2
	Chrysene	2	0.2
	Benzo(b)Fluoranthene	2	0.2
	Benzo(k)Fluoranthene	2	0.2
	Benzo(a)Pyrene	2	0.2
	Indeno(1,2,3-cd)Pyrene	2	0.2
	Dibenzo(a,h)Anthracene	2	0.2
	Benzo(g,h,i)Perylene	2	0.2
		2	0.2
	C_9 - C_{18} Aliphatics	100	20
	C_{19} - C_{36} Aliphatics	100	20
	C_{11} - C_{22} Aromatics	100	20

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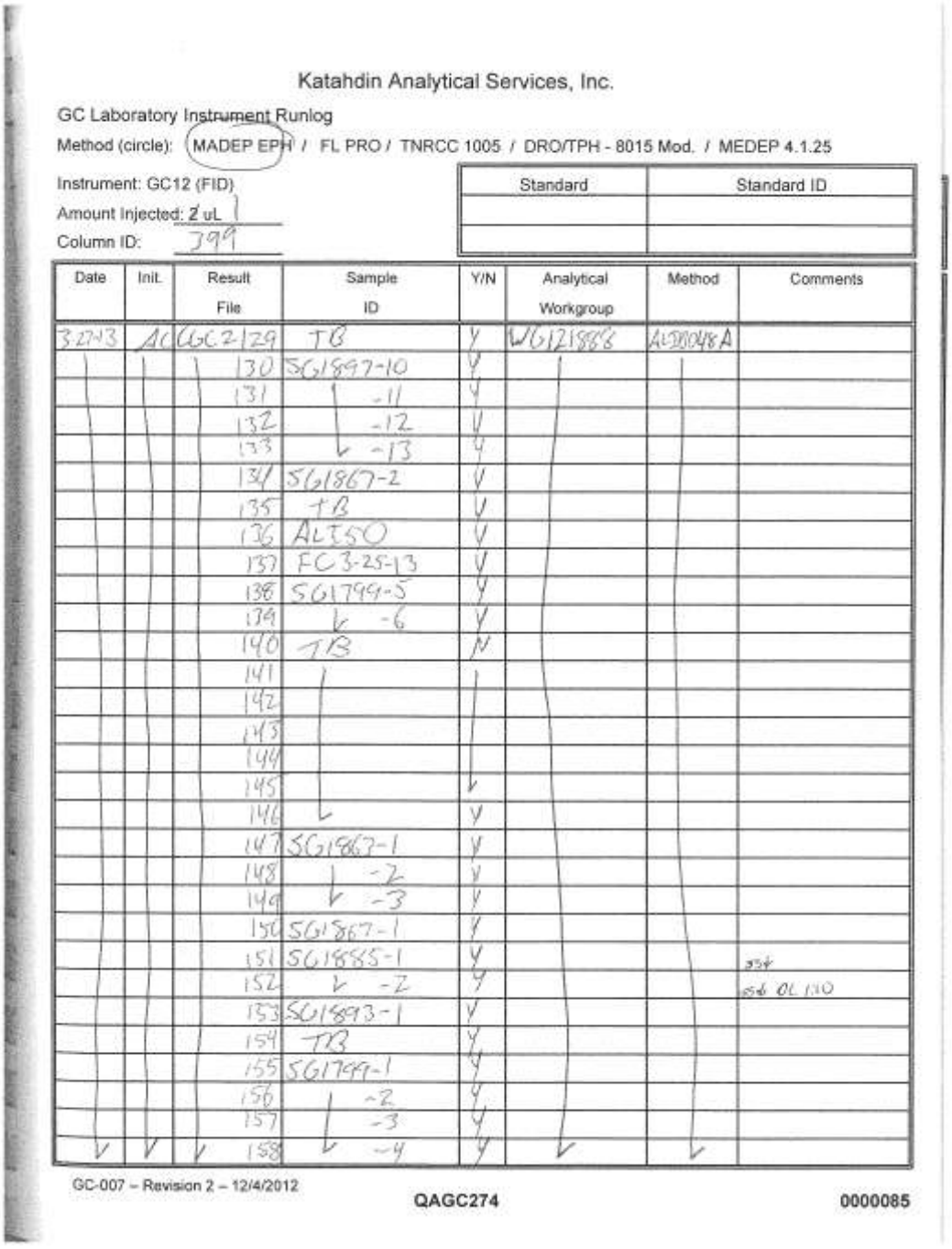
FIGURE 1

EXAMPLE OF MADEP-EPH ANALYTICAL LOGBOOK PAGE



Katahdin Analytical Services, Inc.

GC Laboratory Instrument Runlog

Method (circle): (MADEP EPH) / FL PRO / TNRCC 1005 / DRO/TPH - 8015 Mod. / MEDEP 4.1.25

Instrument: GC12 (FID)
Amount Injected: 2 uL
Column ID: J99

Standard	Standard ID

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
3273	AC	GC2129	TB	Y	WG1218888	AL3048A	
		130	SG1897-10	Y			
		131	-11	Y			
		132	-12	Y			
		133	-13	Y			
		134	SG1867-2	Y			
		135	TB	Y			
		136	ALI50	Y			
		137	FC3-25-13	Y			
		138	SG1799-5	Y			
		139	-6	Y			
		140	TB	N			
		141					
		142					
		143					
		144					
		145					
		146		Y			
		147	SG1867-1	Y			
		148	-2	Y			
		149	-3	Y			
		150	SG1867-1	Y			
		151	SG1885-1	Y			354
		152	-2	Y			354 OL 110
		153	SG1893-1	Y			
		154	TB	Y			
		155	SG1799-1	Y			
		156	-2	Y			
		157	-3	Y			
		158	-4	Y			

GC-007 – Revision 2 – 12/4/2012

QAGC274

0000085

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD

FIGURE 2

EXAMPLE OF HPLC RUNLOG PAGE

Katahdin Analytical Services
HPLC03 Logbook

Analytical Method:	MA-EPH	Other:	Flow: 5.0 mL/min
MeCl2 Lot #: DH997		Hexane Lot #: DH162	

Tray Pos. No.	Sample Identification	Date	Initials	Injection Volume	Dilution	Comments
17	SG 1989-1	4/2/18	JH	1mL	1	
18	Rinse	↓	↓	↓	↓	
1	WG122183-1	4/4/18	JH	1mL	↓	
2	↓ -2					
3	↓ -3					
4	LOD (MW)					
5	LOQ (MW)					
6	SG2112-1					
7	-2					
8	-5					
9	-6					
10	-4					
11	↓ -3					
12	Rinse					
13	↓	↓	↓	↓	↓	
1	Rinse	4-9-18	JMS	1mL	1	
2	WG122104-1					
3	↓ -2					
4	↓ -3					
5	FL					
6	SG21782					
7	SG21783					
8	SG2179-1					
9	SG2258-1					
10	SG2261-1	↓	↓	↓	↓	

Reviewed by: Review Date:

EX-011 – Revision 2 – 03/16/2010

QAEX224

0000071

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD

FIGURE 3

EXAMPLE OF DATA REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Init.* _____) **DueDate**_____ .

Client:	**Verbal Review**	**Primary Review**	**Secondary Reveiw**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT ND's to - PQL* ☐ *MDL* ☐ *LOD* ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitaion Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐) .	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD ○ LAB ○	☐ FORM 6 (s) DOD ○ LAB ○
☐ FORM 4 (s)	☐ FORM 7 (s) DOD ○ LAB ○
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ ○ MDL ○ LOD ○
☐ FORM 8 (s)	☐ Flagging **B** ○ **L** ○ **M** ○ **C** ○
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ ○ MDL ○ LOD ○	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD ○ LAB ○	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD ○ LAB ○	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	Exceedences
< 11	0
11 to 30	1
31 to 50	2
51 to 70	3
71 to 90	4
> 90	5

QA-044 – Revision10 – 04/07/2017

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: ANTHONY BULLENTINI

Review Date: 01 08 20

SOP Number: SA-322-15

SOP Title: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS (MADEP-EPH)

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: [signature] **Date:** 1-9-20

QAO Signature: [signature] **Date:** 01.09.20

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan C. Hughes Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1·22·01	1/22/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5·23·01	5·23·01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 & to Table 2.	DN	5·21·02	5·21·02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB 1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added note that 2 detectors must be used for dual column. Updated method references. Removed calibration and surrogate method mod. from Table 2. Added more info to linear calib. Added extraction references,	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that Surrogates are added to only the aroclor 1660 standards, NOT ALL standards.	LAD	05/09	05/09
10	Revised sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10
12	Removed Sect. 4.5 - Analytical balance. Removed Sect. 5.2.4 - DDT analog standard. Removed Sect. 7.5 - DDT analog standard analysis requirement. Table 1 - Added aver. cal. criteria and corrected CCV & LCS acceptance criteria. Added and removed references to Sect. 10. Updated Figure 2 - data review checklist. Added PCBs 1262 & 1268	LAD	07/11	07/11
13	Added Extraction method 3546. Removed Quickforms references. Added reporting from KIMS. Updated Figures 1 and 2.	LAD	02/13	02/13

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 7 - Added additional information for failing CCVs. Sect. 10 – Updated references. Added Table 3 – DoD QSM 5.0 QC Requirements.	LAD	08/14	08/14
15	Sect. 5 - Added Standards to title. Sect. 7 - Added ICV requirements for all PCBs. Changed 1248 calibration from 6-point to single point. Fixed typos. Rewarded Sect. 7.5.3 for clarification. Sect. 8 - Added MDL/LOD/LOQ information. Updated Fig. 2.	LAD	03/16	03/16
16	Sect. 7 - Added %D Error calculation. Sect. 8 - Added LLOQ reference	LAD	03/17	03/17
17	Sect. 1, 8, 9 and Table 1 - Added LLOQ definition and acceptance criteria. Clarified PQL, LOQ and LLOQ. Updated CCV acceptance criteria. Updated logbook page and data review checklist	LAD	06/17	06/17
18	Sect 7 - Added %Difference and %Drift calculations. Sect. 8 - Added contingency plan. Sect. 10 - Updated references. Removed QSM 4.2 QC Table.	LAD	12/17	12/17
19	Sect. 1 - Updated definitions. Sect 2 - Added GPC Cleanup. Sect. 7 - Edited for clarification and to reflect current practices. Minor edits throughout to reflect current practice and correct typographical errors	LAD	01/19	01/19

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-329-19** titled **ANALYSIS OF AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-329-19** titled **ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Service including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1016 (AR1016), Aroclor-1221 (AR1221), Aroclor-1232 (AR1232), Aroclor-1242 (AR1242), Aroclor-1248 (AR1248), Aroclor-1254 (AR1254), Aroclor-1260 (AR1260), Aroclor-1262 (AR1262) and Aroclor-1268 (AR1268). Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

1.1. Definitions

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term LOQ must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, samples and spiked samples prior to analysis. They are also included in the AR1660 standard used for calibration and the calibration verification standard. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of

the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3640A – Gel Permeation Chromatography (GPC) cleanup, Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Refrigerator for storage of extracts and standards.

5.0 REAGENTS AND STANDARDS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in

standards prep logbook. A separate standard is used for each Aroclor. The concentrations of the working calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The analytes AR1016, AR1260 and the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) are combined into one mixture called AR1660. AR1016 and AR1260 are prepared at the aroclor concentrations above, while the surrogates are at the respective concentrations 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3546 or 3550. Tissue samples are extracted using method 3540 or 3550. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Helium, Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)
Run time: 30 min
Injection size: 2 ul

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of AR1660, AR1242, and AR1254 are prepared and routinely analyzed.

Six-point calibration standards of AR1221, AR1232, AR1248, AR1262 and AR1268 are also prepared and routinely analyzed on the primary PCB instrument. At a minimum, a single point calibration standard is analyzed for these Aroclors. If using a single point and the Aroclor is required for a project and is detected in a sample, then the GC would be calibrated for the Aroclor and the samples would be reanalyzed.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Five characteristic peaks from each Aroclor are used to generate the calibration curve. AR1221 is the exception where only 2 peaks are used. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.1.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.1.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.1.4 A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990.

The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for work originating in their state.

7.3.2.3 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

7.3.3 All six point calibration curves (AR1660, AR1242 AR1454 and if any other Aroclor is required) must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 20%, reanalyze all samples since the last successful calibration verification. A new calibration curve may need to be prepared for that analyte.

The average result for 5 (2 for AR1221) peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM, current version, compliance, if the CCV fails the above criteria and reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ±

0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 Gas chromatographic analysis

7.5.1 Shake samples and let them sit for one minute before vialing for analysis.

7.5.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes. The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

7.5.3 Samples are analyzed in a batch referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards.

When an Initial Calibration is not performed, the calibration must be verified with a Calibration Verification Standard (CV) prior to sample analysis. The calibration for each Aroclor detected in the subsequent analysis must be verified. Typically, for samples whose PCB composition is unknown, CVs for AR1660, AR1242 and AR1254 are analyzed. At a minimum, the AR1660 calibration must be verified because this calibration includes the surrogates.

A CCV must be analyzed at the beginning of every 12-hour clock, after every 10 samples and at the end of the analytical sequence. The CCV concentration should alternate between 1.0 ug/mL and 0.25 ug/mL standards.

The calculated Aroclor concentration in the CV must not exceed a difference (or drift) of ± 20%.

$$\% \text{ Drift} = \frac{\text{Measured Amount} * 100}{\text{True Amount}}$$

Where: Measured Amount = concentration determined by the calibration
True Amount = concentration of the analyte ion the standard

$$\% \text{ Difference} = \frac{CF_v - CF_{Mean} * 100}{CF_{Mean}}$$

Where: CF_v = calibration factor calculated for the calibration verification standard.
CF_{Mean} = mean calibration factor form the initial calibration

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If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected.

If reanalysis cannot be performed, data must be qualified and explained in a narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.5.1.1 If a CV analyzed before or after a batch of samples has a greater than 120% recovery and the analyte was not detected in the associated samples, then reanalysis may not be necessary.

7.5.1.2 If a CV analyzed after a batch of samples has a less than 80% or greater than 120% recovery and the analyte was detected in the associated samples, then reanalysis is necessary to ensure accurate quantitation.

7.5.1.3 If a CV analyzed after a batch of samples has a greater than 120% recovery and the analyte was not detected in the associated samples, then reanlaysis is necessary to ensure that the detector response had not deteriorated to the point that the analyte would not have been detected even though it was present. However, in some cases, where obvious sample matrix carry over is affecting the closing CV, the data may be reported with narration.

7.5.2 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.5.3 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.5.3.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the

height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.5.3.2 Five Aroclor (two for AR1221) concentrations are calculated using the peak heights of the characteristic peaks of the Aroclor. These concentrations are then averaged to determine the concentration of that Aroclor.

7.5.4 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.5.5 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665). **Note:** Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.5.6 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.6 Calculations

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the most recent laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or

a matrix effect, the data is narrated. If the surrogate recoveries are low and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD QSM (current version) use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.
Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 “Manual Integration on GC/MS, GC, HPLC and IC Datasystems” for more information.

7.7.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a “J” indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the other column. In this scenario, the result would be reported from one column and need to be “Q” flagged to indicate that it was not confirmed on a second column.

7.7.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is

provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. The DoD QSM states that a method blank is considered to be contaiminated if the concentration of any analytein the blank exceeds ½ of the LOQ and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.3 LCS, MS/MSD and Surrogate Spike Concentrations and Corrective Actions:

8.3.1 The LCS and the MS/MSD are spiked at the same concentration with AR1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
AR1660	5.0	0.17

8.3.2 The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3.3 LCS and MS/MSD acceptance criteria and Corrective Action:

All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM (current version), use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM (current version), apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be </= 30% between MS and MSD.

8.3.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.4 Non-conformance Report: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report (NCR) must be initiated as soon as possible to document resolution.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target

variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8082A.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8000D

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

Katahdin Analytical Services, SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin Analytical Services, SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	Average Model – at least 5 points, % RSD </= 20% Linear Model – at least 5 points, correlation coefficient (r) ≥ 0.995 Quadratic Model – at least 6 pt calibration, coefficient of determination (r^2) ≥ 0.990 % Error must be ≤ 30% or RSE must be ≤ 20%	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample,5 or 6-pt calibration (depending on calibration model) of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 20 % D	(1) Evaluate the samples: If the %D >+20% and sample results are <PQL, narrate. (2) If %D >±20% only on one channel, narrate. If %D >±20% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL / LLOQ	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Laboratory statistically derived limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.
Sample Duplicate	One sample duplicate per ten samples if requested	RPD ≤20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte</= 20%; Option 2: linear least squares regression for each analyte: r >/= 0.995; Option 3: non-linear least squares regression (quadratic) for each (quadratic) for each analyte: r2 >/= 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD = 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD </= 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

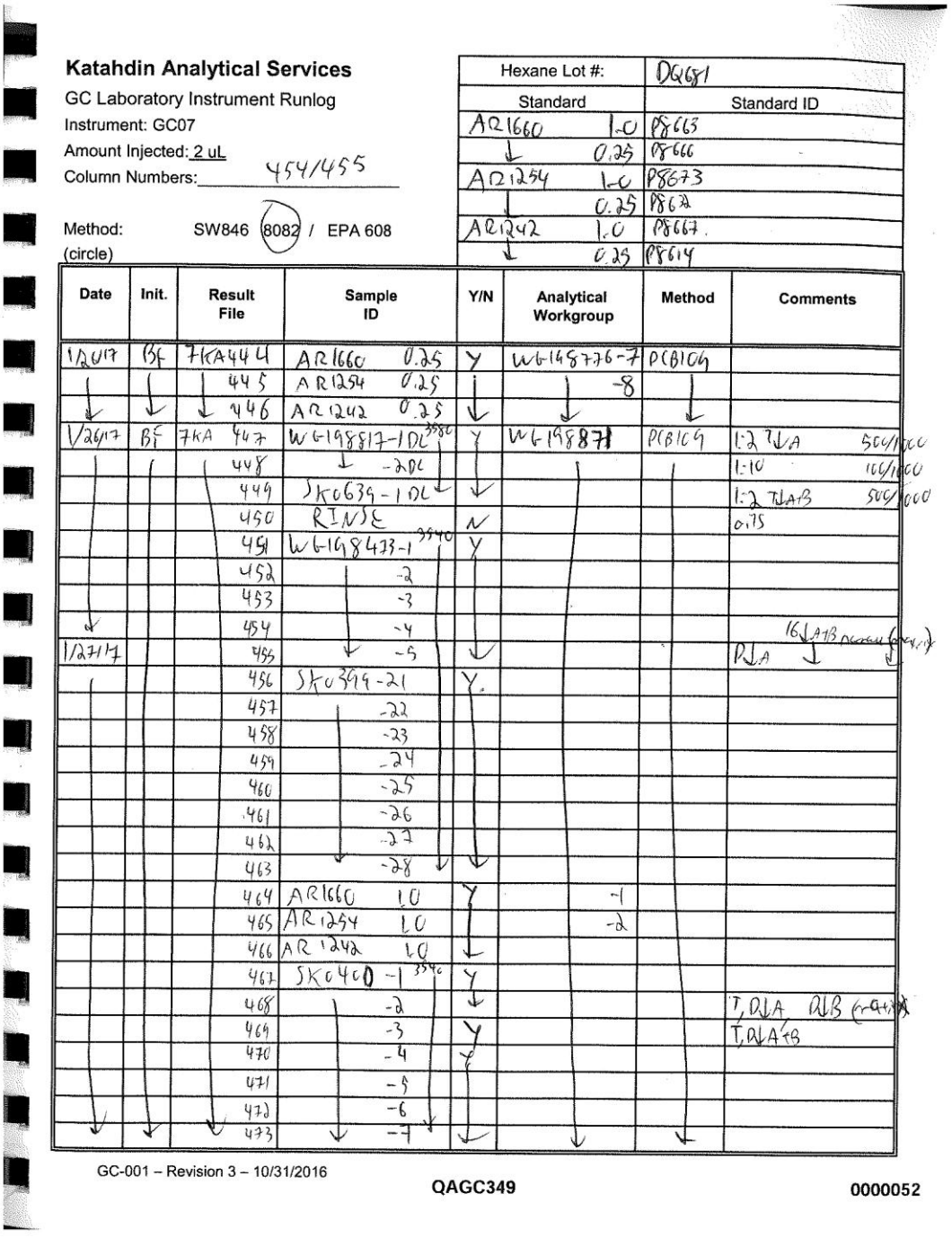
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-19	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG



Katahdin Analytical Services
GC Laboratory Instrument Runlog
Instrument: GC07
Amount Injected: 2 uL
Column Numbers: 454/455
Method: SW846 (8082) / EPA 608
(circle)

Hexane Lot #:	DQ681
Standard	Standard ID
AR1660 1.0	P8663
↓ 0.25	P8666
AR1254 1.0	P8673
↓ 0.25	P8672
AR1242 1.0	P8667
↓ 0.25	P8614

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
1/20/17	BF	7KA444	AR1660 0.25	Y	WG148776-7	PCB104	
↓	↓	445	AR1254 0.25	↓	-8	↓	
↓	↓	446	AR1242 0.25	↓	↓		
1/26/17	BF	7KA 447	WG198817-1DL 3980	Y	WG198871	PCB104	1:2 TLA 500/1000
		448	↓ -2DL				1:10 100/1000
		449	SK0639-1DL	↓	↓	↓	1:2 TLAB 500/1000
		450	RINSE	N			0.75
		451	WG198433-1 3540	Y			
		452	-2				
		453	-3				
↓		454	-4				
1/27/17		455	-5	↓			16 TLAB Accuracy (low, mid) RLA
		456	SK0369-21	Y			
		457	-22				
		458	-23				
		459	-24				
		460	-25				
		461	-26				
		462	-27				
		463	-28	↓			
		464	AR1660 1.0	Y	-1		
		465	AR1254 1.0		-2		
		466	AR1242 1.0	↓			
		467	SK0400-1 3540	Y			
		468	-2	↓			T, DLA, RLB (mid, high)
		469	-3	Y			T, DLA+B
		470	-4	↓			
		471	-5				
		472	-6				
		473	-7	↓	↓	↓	

GC-001 – Revision 3 – 10/31/2016

QAGC349

0000052

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

REVIEW CHECKLIST ☐ Full Package

PRIMARY

Verbal Due Date _____ . (☐ **Verbals Rev. turned in** *DATE:* _____ *Int.* _____) **DueDate**_____.

Client:	**Verbal Review**	**Primary Review**	**Secondary Reveiw**
Method: **Level :**	Date:	Date:	Date:
SDG No.	Initials:	Initials:	Initials:
Login No.			Approved: ☐ Yes

DODQSM (4.2) ☐ **DODQSM (5.0)** ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐

(*REPORT ND's to -* ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐)

	Verbal	Final
Verify the above checked criteria are being used throughout the package.	☐	☐
Verify QC limits and PQLs are correct (LCS, Form 2, Form1)	☐	☐
Merged results (Report single ROA ☐) (Report both ROAs ☐)	☐	☐
Extraction Method and Version & Analysis Method and Version Correct.	☐	☐
Date Sampled, Extracted, Analyzed are correct.	☐	☐
Total Solids is entered on the Quantitaion Report and Form1	☐	☐
Flagging of all ROAs correct (DOD ☐ / Florida ☐).	☐	☐
Manual integrations. Date, Initialed and Coded? (Narrate Level 4 samples only).	☐	☐
Were manual corrections made which may be lost if data needs reprocessing?	☐	☐
Narrate any method deviations. (Blanks, LCS's, ICAL, IND, CCV etc.).	☐	☐
Narrative complete and accurate.	☐	☐
All needed forms & raw data are present & in the correct order in the PDF.	☐	☐
All log book pages included (Runlogs, ICAL pgs, Soil wts, Extr, TCLP, SPLP, grinding & GPC)	☐	☐
Level 3 packages include all three PDF files (SUM , ARC, RAW).	☐	☐
Package PDF's copied to the appropriate To Review folder	☐	☐

Package PDF Requirement Level 3 Reports

SUM - (if all forms) - 1, 2, 3, 4, 5, 6, 7, 8. ☐
ARC - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (samples with raw data), 6, 7. ☐
RAW - 2 ,4, 1 (blks), 3 lcs, 3 msds, 5, 8, 1 (sample ROAs only), 6, IND recoveries, 7. ☐

SECONDARY REVIEW	
☐ FORM 2 (s) DOD O LAB O	☐ FORM 6 (s) DOD O LAB O
☐ FORM 4 (s)	☐ FORM 7 (s) DOD O LAB O
☐ FORM 5 (s)	☐ FORM 1 Sample(s) LOQ O MDL O LOD O
☐ FORM 8 (s)	☐ Flagging B O L O M O C O
☐ FORM 10 (s)	☐ Manual Integrations
☐ FORM 1 Blank (s) LOQ O MDL O LOD O	☐ Logbook Pages
☐ FORM 3 LCS/LCSD (s) DOD O LAB O	☐ Chromatograms & RTs
☐ FORM 3 MS/MSD (s) DOD O LAB O	☐ Manual changes rechecked if data reprocessed

DOD	
CMPD List	**Exceedences**
< 11	**0**
11 to 30	**1**
31 to 50	**2**
51 to 70	**3**
71 to 90	**4**
> 90	**5**

QA-044 – Revision10 – 04/07/2017

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
AR1016	0.50	17
AR1221	0.50	17
AR1232	0.50	17
AR1242	0.50	17
AR1248	0.50	17
AR1254	0.50	17
AR1260	0.50	17
AR1262	0.50	17
AR1268	0.50	17

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-500
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 10/23/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 ~~02~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example. Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS manual. Added CA-108 reference for subsampling	LAD	02/09	02/09
07	Removed targeting sample weights. Minor changes to section 7. Updated logbook example.	LAD	08/10	08/10
08	Section 5 - Removed baking and rinsing Na_2SO_4, added pH determination for MeCl$_2$, added solvent lot check for acetone. Section 7 - Added information for alternative final volumes, changed spike addition from before Na_2SO_4 to after, added MS/D criteria. Minor changes to reflect current practice.	LAD	03/12	03/12
09	Sect. 4 - Updated Sonicator make and model. Sect. 7 - Changed where hexane is added for solvent exchange. Added decanting information. Updated Sonicator setup. Updated Figure 1 - logbook example.	LAD	04/14	04/14
10	Sect. 5 - Added extra LCS Compound lists for project specific pesticides/PCBs. Sect. 7 - Updated procedures, Updated sonication instructions. Removed references to old Sonicator, Mentioned additional LCSs, Removed redundancies. Minor formatting changes and grammatical corrections throughout. Updated figure 1 - logbook example	LAD	09/17	09/17
11	Added PCB Congener information. Added wooden tongue depressors. Updated references.	LAD	03/19	03/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-500-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should

also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest

the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors with 85 mm plate diameter (or equivalent)

4.6 Sonicator – Ultrasonic Processor – Qsonica model Q500 or equivalent

4.7 Spatula - stainless steel

4.8 Wooden Tongue Depressors

4.9 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.10 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.11 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.12 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.13 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.14 Balance – top-loading, capable of weighing to 0.01 g

4.15 Nitrogen evaporation apparatus

4.16 Vials (12 mL, 4 mL, 2 mL, and/or 1.8 mL) screw cap vials with Teflon-lined caps or Teflon/silicon septum caps (new, certified clean by manufacturer)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

5.0 REAGENTS AND STANDARDS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC/MS analysis. Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer. The pH must be > 5.

5.4 Acetone and hexane - pesticide grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC analysis.

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 PCB Congener Surrogate Spiking Solution: Prepare a solution of Tetrachloro-m-xylene (TCX), 2,2',4,5',6-pentachlorobiphenyl (PCB 103) and 2,3,3',4,5,5',6-heptachlorobiphenyl (PCB 192) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix spike/Lab control sample spiking solutions:

5.8.1 For every extraction batch, prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
alpha-Chlordane	0.5

beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
gamma-Chlordane	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5

5.8.2 Extra Pesticide Matrix spike/Lab control sample spiking solutions: The following pesticides may be required for certain projects; if so, prepare a spiking solution in pesticide grade methanol that contains the relevant target analytes listed below:

Analyte	**ug/mL**
2,4'-DDD	0.5
2,4'-DDE	0.5
2,4'-DDT	0.5
Alachlor	10.0
Chlorpyrifos	0.5
Dichlobenil	0.5
Hexachlorobenzene	0.5
Mirex	0.5
cis-Nonachlor	0.5
trans-Nonachlor	0.5
Oxychlordane	0.5

5.8.3 Multi-Response Pesticide Matrix spike/Lab control sample spiking solutions: The following multi-response pesticides may be required for certain projects; if so, prepare a separate spiking solution in pesticide grade methanol for each required multi-response target analyte at the concentrations listed below:

Analyte	**ug/mL**
Toxaphene	10.0
Technical Chlordane	10.0
Chlorothalonil	0.5

Propiconizole (Tilt)	0.5

5.9 PCB Arochlor Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260, both at 5.0 ug/mL. For DoD QSM 5.0 or 5.1 projects, additionally prepare spiking solution in pesticide grade acetone that contains PCB Arochlor 1254 at 5.0 ug/mL.

Analyte	**ug/mL**
Arochlor 1016	5.0
Arochlor 1260	5.0
Arochlor 1254	5.0

5.10 PCB Congener Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in pesticide grade acetone that contains all target analytes listed below:

ANALYTE	**ug/mL**
PCB 8 – 2,4'-dichlorobiphenyl	0.2
PCB 18 – 2,2',5-trichlorobiphenyl	0.2
PCB 28 – 2,4,4'-trichlorobiphenyl	0.2
PCB 44 – 2,2',3,5'-tetrachlorobiphenyl	0.2
PCB 49 – 2,2',4,5'-tetrachlorobiphenyl	0.2
PCB 52 – 2,2',5,5'-tetrachlorobiphenyl	0.2
PCB 66 – 2,3',4,4'-tetrachlorobiphenyl	0.2
PCB 77 – 3,3',4,4'-tetrachlorobiphenyl	0.2
PCB 81 – 3,4,4',5-tetrachlorobiphenyl	0.2
PCB 87 – 2,2',3,4,5'-pentachlorobiphenyl	0.2
PCB 101 – 2,2',4,5,5'-pentachlorobiphenyl	0.2
PCB 105 – 2,3,3',4,4'-pentachlorobiphenyl	0.2
PCB 114 – 2,3,4,4',5-pentachlorobiphenyl	0.2
PCB 118 – 2,3',4,4',5-pentachlorobiphenyl	0.2
PCB 123 – 2,3',4,4',5'-pentachlorobiphenyl	0.2
PCB 126 – 3,3',4,4',5-pentachlorobiphenyl	0.2
PCB 128 – 2,2',3,3',4,4'-hexachlorobiphenyl	0.2
PCB 138 – 2,2',3,4,4',5'-hexachlorobiphenyl	0.2
PCB 153 – 2,2',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 156 – 2,3,3',4,4',5-hexachlorobiphenyl	0.2
PCB 157 – 2,3,3',4,4',5'-hexachlorobiphenyl	0.2
PCB 167 – 2,3',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 169 – 3,3',4,4',5,5'-hexachlorobiphenyl	0.2
PCB 170 – 2,2',3,3',4,4',5-heptachlorobiphenyl	0.2
PCB 180 – 2,2',3,4,4',5,5'-heptachlorobiphenyl	0.2

PCB 183 – 2,2',3,4,4',5',6-heptachlorobiphenyl	0.2
PCB 184 – 2,2',3,4,4',6,6'-heptachlorobiphenyl	0.2
PCB 187 – 2,2',3,4',5,5',6-heptachlorobiphenyl	0.2
PCB 189 – 2,3,3',4,4',5,5'-heptachlorobiphenyl	0.2
PCB 195 – 2,2',3,3',4,4',5,6-octachlorobiphenyl	0.2
PCB 206 – 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	0.2
PCB 209 – 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	0.2

5.11 Store the solutions at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

The holding time for extraction of Pesticide sediment/soil samples by Method 3550 is 14 days from date of sample collection.

The holding time for PCB only sediment/soil is 30 days. SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit.

Samples extracted for both Pesticide and PCB must be extracted within the 14 day hold time.

Analysts should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction and Analytical methods
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sonicator Amplitude
- Sample pH (if applicable)

- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weights
- Surrogate and spike amounts
- Boiling chip date
- Balance ID
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- Lot numbers of the vials the concentrated extracts are stored in.

EXTRACTION OF LOW LEVEL SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of approximately 30.0 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

7.1 Do not decant any water on the sediment sample.

Note: Some work orders may specify to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2 Mix with a stainless steel spatula or tongue depressor to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“, for more detailed guidance on subsampling to ensure reproducibility.**

7.3 Weigh out approximately 30.0 g plus (> 30 g) portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Refer to section 7.7 for surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or tongue depressor. Keep the spatula/depressor in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out 30.0 ± 0.05 g of purified sand in a labeled 400 mL beaker. Refer to section 7.7 for surrogate addition instructions. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out 30.0 ± 0.05 g of purified sand into a labeled 400 mL beaker. Refer to sections 7.7 and 7.8 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. Additional LCS's for project specific compounds may also be required. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximately 30.0 g plus (>30 g) portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.7 and 7.8 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

Note: A MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. If samples are to be concentrated to a lower than 10 mL final volume the surrogate

spike must be adjusted so the final concentration in the samples is 0.10 ug/mL. The surrogate spike should be added after the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.8 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. If samples are to be concentrated to a lower than 10 mL final volume the surrogate spike must be adjusted so the final concentration in the samples is 0.5 ug/mL for pesticides and 5 ug/mL for PCBs. The LCS/MS spike should be added after the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.9 To each mixed and spiked sample, blank, LCS/LCSD, and/or MS/MSD in the extraction batch, add approximately 100 mL of the 1:1 methylene chloride/acetone solution.

7.10 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula/depressor to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.11 Sonicator settings:

7.11.1 Set pulse timer to 1 ½ minutes.

7.11.2 Set pulse to 1 and 1. This sets the sonicator to pulse for 1 second and rest for 1 second, with the timer set for 1 ½ minutes this equals a 3 minute total sonication.

7.11.3 Set amplitude to 40%. Record this in the logbook.

These settings are stored in the unit and do not have to be entered with each use.

7.12 Extraction by sonication:

7.12.1 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.12.2 Turn sonicator on and allow 3 minutes for sonication process.

7.12.3 Refer to the Operating Manual for further information.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse flask, funnel, and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat steps 7.9, 7.10, 7,12, and 7.13 two additional times, each time adding approximately 100 mL portions of 1:1 methylene chloride:acetone to the beaker. Before each sonication, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula/depressor. Use the same Buchner funnel and filter for all three sonications for each sample. Decant the extraction solvent into the Buchner funnel after each sonication. On the final (third) sonication, pour the entire beaker contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract.

7.15 Repeat the extraction (steps 7.9 through 7.14) for each sample in the extraction batch.

CONCENTRATION OF LOW LEVEL EXTRACTS FOR PESTICIDES/PCBs

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, proceed to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. PCB Congener samples must be GPC'd. If samples are not to be GPC'd, proceed to step 7.19.

7.19 For a solvent exchange, add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39 ºC for methylene chloride).. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap.

7.24 Complete quantitative transfer of the extract to a vial by using hexane: Add hexane to adjust the volume of the hexane extract to the appropriate final volume, in a 12 mL vial (for 10 mL or 5 mL final volumes), a 4 mL vial (for 2 mL final volume), or a 1.8 mL vial (for 1 mL final volume). Rinse sides of tube with hexane and transfer rinses to final vial, taking care not to exceed the intended final volume. Use the appropriate reference vial for samples with 10, 5, 2 or 1 mL final volumes.

7.25 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.26 All sample extracts for 8082 PCB Arochlor analysis that are not GPC’d must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA-525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other

corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

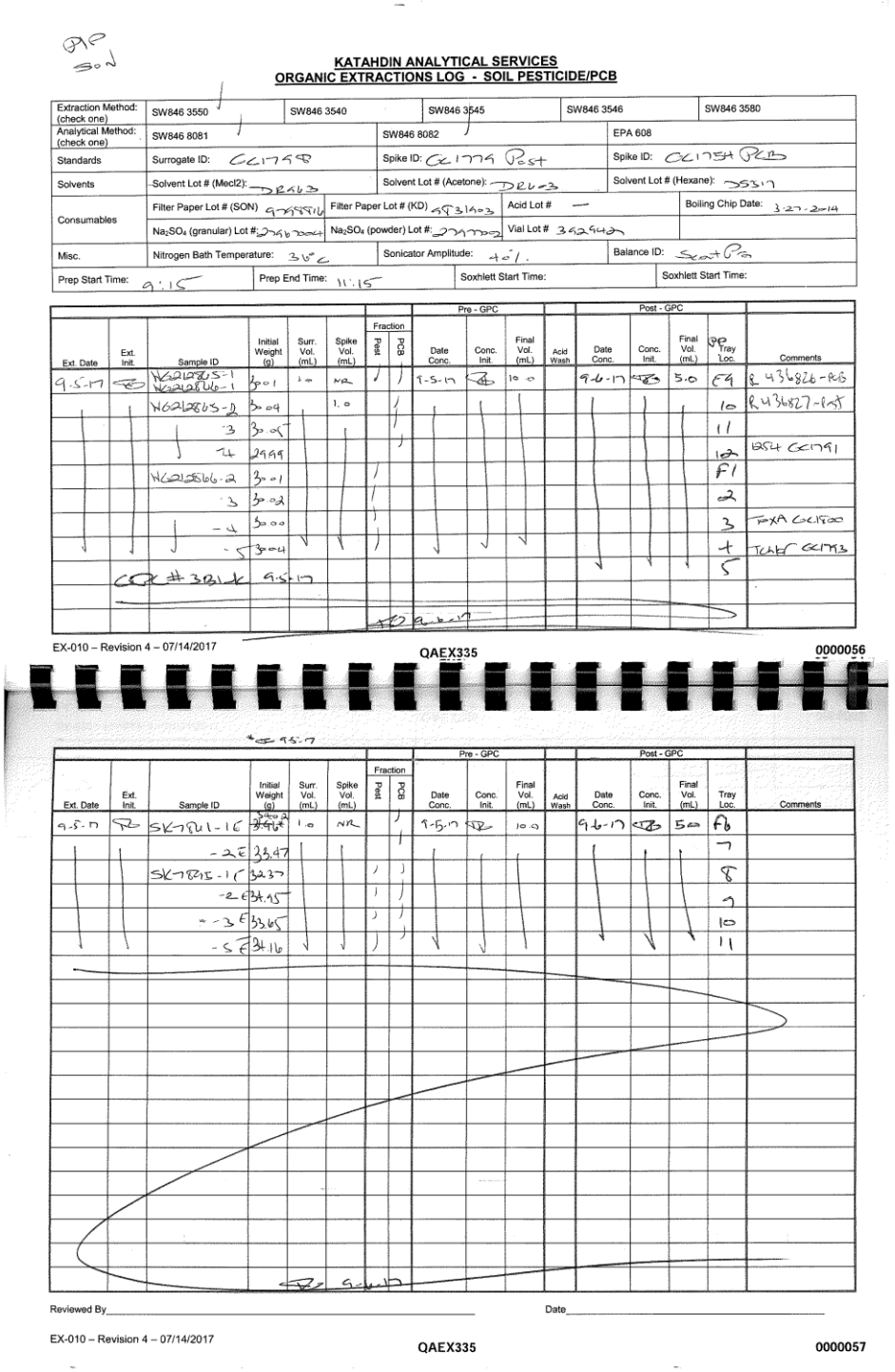
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-11	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550	SW846 3540	SW846 3545	SW846 3546	SW846 3580
Analytical Method: (check one)	SW846 8081	SW846 8082		EPA 608	
Standards	Surrogate ID: CC1749	Spike ID: CC1774 Pest		Spike ID: CC1754 PCB	
Solvents	Solvent Lot # (MeCl2): DR663	Solvent Lot # (Acetone): DR663		Solvent Lot # (Hexane): 35317	
Consumables	Filter Paper Lot # (SON) 9498716	Filter Paper Lot # (KD) 9431403	Acid Lot # —	Boiling Chip Date: 3-27-2014	
	Na_2SO_4 (granular) Lot #: 2916204	Na_2SO_4 (powder) Lot #: 2917002	Vial Lot # 3529432		
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Amplitude: 40%		Balance ID: Scout Pro	
Prep Start Time: 9:15	Prep End Time: 11:15	Soxhlett Start Time:		Soxhlett Start Time:	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Pest	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Tray Loc.	Comments
9.5.17	RP	HG21[illegible]-1 HG21[illegible]-1	30.01	1.0	NR	✓	✓	9-5-17	RP	10.0		9-6-17	[illegible]	5.0	F9	R 436826-PCB
		HG21[illegible]-2	30.04		1.0	✓	✓	✓	✓	✓		✓	✓	✓	10	R 436827-PAT
		-3	30.05		✓	✓	✓	✓	✓	✓		✓	✓	✓	11	
		-4	29.99		✓	✓	✓	✓	✓	✓		✓	✓	✓	12	1254 CC1791
		HG21[illegible]-2	30.01		✓	✓	✓	✓	✓	✓		✓	✓	✓	F1	
		-3	30.02		✓	✓	✓	✓	✓	✓		✓	✓	✓	2	
		-4	30.00		✓	✓	✓	✓	✓	✓		✓	✓	✓	3	TCMX CC1720
		-5	30.04		✓	✓	✓	✓	✓	✓		✓	✓	✓	4	TCMX CC1793
		CCV #3814	9.5.17												5	

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*RP 9.5.17

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Pest	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Tray Loc.	Comments
9.5.17	RP	SK7821-1E	30.96 Spike 2	1.0	NR	✓		9-5-17	RP	10.0		9-6-17	[illegible]	5.0	F6	
		-2E	33.47												7	
		SK7821-1E	32.37			✓	✓	✓	✓	✓		✓	✓	✓	8	
		-2E	34.95			✓	✓	✓	✓	✓		✓	✓	✓	9	
		-3E	33.65			✓	✓	✓	✓	✓		✓	✓	✓	10	
		-5E	34.16			✓	✓	✓	✓	✓		✓	✓	✓	11	

Reviewed By_____ Date_____

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ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Melissa Rossi

Review Date: 1-7-20

SOP Number: CA-500-11

SOP Title: Preparation of sediment/soil samples by sonication using method 3550 for subsequent pesticides/PCBs analysis

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-29-2020

QAO Signature: Lucia Dimond **Date:** 020520

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial Sample volume. Added extracted Sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial Volume determination. Added Reference to CA-109. Updated logbook example. Added if extract goes dry - reextract.	LAD	10/09	10/09
07	Sect. 5 - Removed baking and rinsing $NaSO_4$. Added 1,4-Dioxane to SIM Surrogate Mix. Sect. 7 added acid to B/N SIM., removed to let separate for 10 minutes minor edits throughout.	LAD	03/12	03/12

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08	Removed Sect. 7.1.9, determining the sample initial volume. Sect. 7.1.4 has this information. Figures 1 and 2 updated.	LAD	05/13	05/13
09	Sect. 5 - Updated prep of SIM/scan Surrogate mix. Sect. 7 - Updated Surrogate spiking directions. Updated Figure 1.	LAD	06/14	06/14
10	Sect. 7 - For separatory funnel, corrected extraction sequence (acid then basic), and that the pH is determined after first shake. Updated Solvent Lot Check Form. Changed KAS INC → KAS	LAD	08/15	08/15
11	Change order of pH checking and spike stds addition. Replace use of HCL with H_2SO_4 to adjust pH. Added test for residual chlorine. Updated method references for NELAC, DOD + SW 846.	LAD	09/17	09/17
12	Sect. 1 - Corrected order of pH extraction. Sect 7 - Updated for current practice. Sect. 10 - Updated references. Updated logbook page example.	LAD	03/19	03/19

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-12**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-12**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste

stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by separatory funnel and CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a separatory funnel or a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

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4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS AND STANDARDS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC and/or GC/MS analysis.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	20 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.3 Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutrals and 4.0 ug/mL for acids. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem.

5.5.4 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Potassium iodide starch paper

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

Follow the proper procedures for maintaining Internal Chain of Custodies for samples when removing and replacing samples in storage locations. This procedure is described in KAS SOP SD-902, “Sample Receipt and Internal Control”, current revision.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order

consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS).

7.1.3.1 To prepare method blank, add 1 L reagent water to a sample bottle. Pour this into the CLLE body. Be sure that no water leaks into the round bottom flask. Repeat for the LCS.

7.1.3.2 If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis.

7.1.3.3 This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 The initial volume of a sample is determined by comparing the meniscus of the sample to a reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.5.1 Transfer the sample to a CLLE body slowly, being sure that no water leaks into the round bottom flask.

7.1.5 To prepare a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

REMEMBER: If both SCAN and SIM analysis are required, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.1.7 To LCS/LCSD and MS/MSD add 1.0 mL of the appropriate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.7.1 For SVOA Scan Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2).

7.1.7.2 For SIM Analysis - add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.3).

7.1.7.3 For SVOA Appendix IX Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2) and 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.4).

7.1.8 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.1.9 For each blank, LCS, MS and sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.10 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.11 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.12 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.13 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.14 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.11 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered with foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the “N-Hi” satellite.

7.1.12 Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples may be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent.

7.2.3.1 To prepare method blank, add 1 L reagent water to a sample bottle. Pour this into the separatory funnel. Repeat for the LCS.

7.2.3.2 If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis.

7.2.3.3 The blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use. (sect. 5.5.2). **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL of the appropriate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 For SVOA Scan Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2).

7.2.7.2 For SIM Analysis - add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.3).

7.2.7.3 For SVOA Appendix IX Analysis - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.2) and 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.4).

7.2.8 For each blank, LCS, MS and sample, rinse the original sample container, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH ≤ 2 with 1:1 H_2SO_4 after addition of surrogates and spikes. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.2.10 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes.

7.2.11 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.2.12 Allow phases to separate. Drain the methylene chloride layer into an amber collection bottle.

7.2.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.15 Repeat the extraction for a third time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.16 Following the third shake, adjust the pH to $\geq$ 11 with 10N NaOH. Add enough 10N NaOH to adjust the pH to $\geq$ 11.

7.2.17 Add 60 mL methylene chloride to each separatory funnel and extract the samples in the same manner described in 7.2.11 – 7.2.14. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.18 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≥ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.19 Repeat 1 more time with 1 more 60 mL aliquot of methylene chloride. Collect the methylene chloride layer in the same amber collection bottle

7.2.20 Sample waste should be poured into the “N-Hi” satellite.

7.2.21 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

7.3.1 For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.2 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts. Record the lot numbers for filter paper, sodium sulfate crystals and methylene chloride in the extractions logbook.

7.3.3 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to

complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.4 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.5 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.6 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.7 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.8 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.9 Transfer all of the extract to a 1.8 mL screw cap vial. Using methylene chloride, adjust the final volume of each extract to 1 mL by comparison to an appropriate reference vial.

Store in refrigerator until GC/MS analysis.

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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:
- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:
- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 3510 and 3520, current revisions.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-12	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-12	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE

S/S
Sep

KATAHDIN ANALYTICAL SERVICES, LLC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3510 (SEP) ✓	SW846 3520 (CLLE)	SW846 3535 (SPE)
Analytical Method:(check one)	SW846 8270 ✓	SW846 8270 SIM ✓	EPA 625 (mark meniscus w/ marker to determine IV)
Surrogate ID: SV2885	Surrogate ID:	Spike ID: SV2869 SV	Spike ID: SV2887 SIM
Methylene Chloride Lot #: DV249-US	pH Paper Lot # HC857466	KI Starch Paper ID: 082117	Note samples requiring TRC neutralization in comments section.
pH (1st Extraction) ≤2	H_2SO_4 Lot # RE0334	pH (2nd Extraction) ≥11	NaOH Lot # 184664
$NaSO_4$ Lot # RE5341	Filter Paper Lot #: 1684 3524	Boiling Stones ID: 3-27-2014	Vial Lot #: 403039
Nitrogen Bath Temperature: 36°C			
Prep Start Time: 9:00	Prep End Time: 11:30	CLLE Start Time: —	CLLE End Date & Time: —

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location SV/MS	Initials	Comments
12-26-18	AC	WG243501-1 WG243502-1	1000	1mL	NR	✓	✓	1mL	12-26-18	A1	JMS	SV- R497250
		WG243501-2	↓		1mL	✓				2		SIM- R497251
		-3	1060			✓				3		MS TL2690-1F1
		-4	1060			✓				4		MSD -1I1
		-5	200		NR	✓				5		diluted to 1000 PBT Blank 1498
		WG243502-2	1000		1mL		✓			6		

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location SV/MS	Initials	Comments
12-26-18	AC	TL2607-1	200	1mL	NR	✓		1mL	12-26-18	A7	JMS	TCLP diluted to 1000
		TL2659-1								8		
		-2								9		
		-3								10		
		TL2661-1	↓							B1		↓
		TL2690-1 B1	1060							2		ms/msd
		-2K	1030							3		
		-3L	1050							4		
		-4J	1060							5		
		-5J	1060							6		
		-6K	1060							7		s.de 4e/a
		TL2691-27A	1030				✓			8		

Reviewed By _____ Date _____

EX-007 – Revision 5 – 11/08/2017 QAEX367 0000117

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT LOT CHECK LOGBOOK

KATAHDIN ANALYTICAL SERVICES

SOLVENT LOT CHECK

SOLVENT:_____

LOT#:_____

DATE RECEIVED:_____

DATE CONCENTRATED:_____

CONCENTRATED BY:_____

PREP METHOD:_____

TRAY LOCATION:_____

ANALYZED BY:_____

PASS/FAIL:_____

EX-027 – Revision 1 – 08/15/2015

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Melissa Rosa

Review Date: 1-5-20

SOP Number: CA-502-12

SOP Title: Preparation of Aqueous Samples of extractable semivolatile analysis.

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-29-20

QAO Signature: Leslie Dimmer **Date:** 02052020

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-510
Revision History
Cover Page
Page 1

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 02/01/01

Operations Manager: John C Buxton Date: 2/2/01

QA Officer: Deborah J. Nadeau Date: 2.1.01

General Manager: Deborah F. Kuhfahl Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Changed figures, inserted database references. Format changes, added pollution prevention.	DN	2.1.01	2/1/01
02 1311	modified to reflect change from TCLP database to handwritten logbooks. Changed metals spiking instructions	LAD	030805	030805
03	Added expiration dates for TCLP fluids (1YR) Added DOC requirement Revised TCLP logbook to include SPLP and spaces for pH and exp. dates.	LAD	01/07	01/07
04	Sect. 4: Added use of fluorinated extraction vessels for organics. Updated TCLP/SPLP Logbook example.	LAD	03/08	03/08
05	Updated Figure 8 - TCLP Extraction logbook page.	LAD	03/09	03/09

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated and/or added references to sections 7, 9 and 10. Updated Figure 8- Logbook page.	LAD	06/10	06/10
07	Revised Logbook and updated Figures 8 and 9. Updated text references to logbook in SOP. Revised section 7.4.2 to require pH measurement by meter rather than pH strips. Added and updated references in section 10	LAD	04/12	04/12
08	Sect. 4 & 5 - Changed reagent and preparation from 1N HNO_3 to 5% HNO_3. Table 2 - Added HNO_3 concentration. Sect. 10 - Added and updated references. Updated Figures 6 → 9	LAD	06/14	06/14
09	Sect. 7 - Added room temperature criteria. Fixed typographical errors. ~~KAS → K~~ KAS INC → KAS throughout LAD 08/15/15	LAD	08/15	08/15
10	Sect. 10 - updated method references. Sect. 4 - added thermometer with min/max temp + use extraction case. Sect. 8 - added MS/MSD per PBT lot. Updated figures 6, 7 & 8 Removed figure for Rotator temp book.	LAD	09/17	09/17
11	Sect. 4. Added smaller filtering apparatus may be used for metals only TCLPs. Updated references. Updated Logbook example.	LAD	07/19	07/19

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-510-11,** titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-510-11,** titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services personnel for TCLP extraction of samples for inorganic and non-volatile organic components using USEPA Method 1311 (Test Methods for Evaluating Solid Waste, Physical / Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The TCLP (Toxicity Characteristic Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of the liquid fractions of the TCLP extract indicates that a regulated compound is present at a concentration that, after accounting for dilution from the other fractions of the extract, would be equal to or above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract. The regulated toxicity characteristic analytes are listed in Table 3.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in TCLP extractions. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in TCLP extractions to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be aware of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method may not be precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes from TCLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Management Plan and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. The composition of the extraction fluid employed depends on the alkalinity of the solid phase of the waste. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed

together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of TCLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the TCLP procedure and are discussed in detail in Section 8.0, Quality Control.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30±2 revolutions per minute (rpm) – see Figure 1. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook (see Figure 7). If the measured rotation rate of an extractor is outside the range 30±2 rpm, it must be taken out of service until it can be repaired.

4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated polyethylene if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.

4.3 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.3.1 For Metals only – A Sidearm Erlemeyer flask attached to vacuum pump may be used.

4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 µm, 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 5% HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.

4.4.1 For Metals Only - a 47 mm glass fiber filter is used.

4.5 pH meter accurate to ±0.05 units at 25°C. The pH meter must be calibrated on each day of use.

4.6 pH indicator strips covering the pH range 0 - 14 in increments of 1 pH unit.

4.7 Laboratory balance accurate to within ±0.01 grams (all weight measurements are to be within ±0.1 grams).

4.8 Beakers flasks, glass, 500 mL..

4.9 Watch glasses, appropriate diameter to cover beakers.

4.4 Magnetic stirrer.

4.5 Themometer, capable of reading min/max temperature

5.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Laboratory reagent grade water – Water free of any analyte of interest. Laboratory reagent grade water should be monitored periodically for impurities.

5.2 Hydrochloric acid, concentrated (HCl) – reagent grade.

5.3 Nitric acid, concentrated (HNO_3) – reagent grade.

5.4 Hydrochloric acid, 1N. Dilute 83 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.

5.5 Nitric acid, 5%, for acid-washing filters. Dilute 500 mL reagent grade HNO_3 to 10 L with laboratory reagent grade water.

5.6 Sodium hydroxide (NaOH) – reagent grade, pellets.

5.7 Glacial acetic acid (CH_3COOH) – reagent grade.

5.8 Extraction Fluid #1 - Add 114 mL glacial acetic acid and 51.4 g sodium hydroxide to approximately 1500 mL of laboratory reagent grade water in a clean borosilicate glass extraction vessel reserved for this purpose. Shake until the sodium hydroxide is

completely dissolved. Pour this solution into a clean, graduated 20 L carboy reserved for Extraction Fluid #1 and rinse the extraction vessel three times with approximate liter volumes of laboratory reagent grade water, adding the rinsates to the carboy. Add laboratory reagent grade water to the carboy to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 4.93 ±0.05. The fluid may be used for up to one year from the preparation date.

5.9 Extraction Fluid #2 - Add approximately 10 L of laboratory reagent grade water to a graduated 20 L carboy reserved for Extraction Fluid #2. Add 114 mL glacial acetic acid to the carboy, and then add laboratory reagent grade water to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 2.88 ±0.05. The fluid may be used for up to one year from the preparation date.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile TCLP Extraction Logbook (Figure 7) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the TCLP Fluid Preparation and Use Logbook (Figure 6). Upon preparation, each new batch of extraction fluid is assigned a 3-digit batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the TCLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C and opened immediately prior to TCLP extraction.

6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

6.4 Sample holding times for non-volatile TCLP extraction and analysis summarized in the following table:

TCLP PARAMETER	FROM COLLECTION TO TCLP EXTRACTION	FROM TCLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 2 and 3. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the TCLP extraction, Section 7.4.

All information and measurements pertaining to TCLP extractions are recorded in the Non-Volatile TCLP Extraction Logbook (Figure 7). In the following procedure, the section or column of the Non-Volatile TCLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Column C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section III**) - Percent solids is defined for TCLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.

If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Column A**) and the container that will receive the filtrate (filtrate vessel) (**Column B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.3 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the weigh boat and waste (**Column C**).

7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.1.6 Weigh the weigh boat and any residue clinging to it (**Column D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column E**).

7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.1.8 Weigh the filtrate vessel and its contents (**Column F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column G**).

7.1.9 Calculate the percent wet solids as follows (**Column H**):

$$\text{Percent wet solids} = \frac{\text{(Total weight of waste)-(Weight of liquid phase)}}{\text{Total weight of waste}}$$

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7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100± 20°C until two successive weighings yield the same value within ±1%. Record the weight of the filter and dry solids (**Column I**).

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Column J**).

7.2.4 Calculate the percent dry solids as follows (**Column K**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile TCLP Extraction Logbook (e.g. “No apparent solids present – dry solid weight is due to entrained non-volatile liquid”), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm^3. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a

surface area or particle size as described above. Perform particle size reduction on the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the particle size (if necessary) to approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.4.2 Add 96.5 mL of laboratory reagent grade water to the beaker, cover with a watch glass, and stir vigorously for 5 minutes using a magnetic stirrer. Using the pH meter, measure and record the pH to at least one decimal place(**Section II**). If the pH is <5.0, use Extraction Fluid #1 and proceed with the TCLP extraction, Section 7.5.

7.4.3 If the pH from Section 7.4.2 is >5.0, add 3.5 mL 1N HCl, stir briefly, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.

7.4.4 Let the solution cool to room temperature and record the pH (**Section II**). If the pH is <5.0, use Extraction Fluid #1. If the pH is still >5.0, use Extraction Fluid #2. Proceed to the TCLP extraction, Section 7.5.

TCLP EXTRACTION FOR NON-VOLATILES

7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis. Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for information on subsampling.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 g minimum), record the weight (**Section II**), and proceed to Section 7.5.11. If the sample is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Section IV**, **Column L**).

7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.

7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Column M**). If the waste contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing >0.5% dry solids, information is obtained in Section 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.

7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.5.7 Weigh the weigh boat and any residue clinging to it (**Column N**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column O**).

7.5.8. Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing as moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is reached under 10 psi, and if no additional liquid has passed through he filter in any 2-minute interval, slowly increase in pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has creased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.5.9 Weigh the filtrate vessel and its contents (**Column P**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column Q**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Column R**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Column S**).

7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.3. Describe the particle size reduction process in the Comments section (**Section V**) of the logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{(20)\,(\text{Weight of wet solids})}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID, the amount used, and the pH (measured on day of use) in **Sections I and II** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30± 2 RPM during the extraction period of 18 $\pm$ 2 hours at 23 ± 2 ºC. Record the extraction start and end times and the room temperatures (min/max throughout extraction process) in **Section I** of the logbook. In order to maintain the required temparature range throughout the extraction, a temperature controlled exctraction case is used that has temperature maintained by an individual heating and cooling system.

NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Section 7.5.6. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

7.5.14 Prepare the TCLP extract as follows:

7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the TCLP extract. Proceed to Section 7.5.15.

7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the TCLP extract. Proceed to Section 7.5.15.

7.5.14.3 If the initial liquid phase of the waste, as obtained from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in the Comments section (**Section V**) of the logbook. Individually analyze these two liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.6.

7.5.15 Following collection of the TCLP extract, the pH of the extract should be measured and recorded (**Section II**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. All other aliquots must be stored under refrigeration (4°C) until analyzed.

7.6 The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in

those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.7 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 1311 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of

the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel. .

8.1.1 After TCLP extraction, TCLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Section 6.4). For this reason it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for TCLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 300. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full TCLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 300. Because the holding time for the previous TCLP method blank for pesticides and herbicides has expired, a new TCLP method blank must be extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 300 has already been analyzed for these constituents.

8.1.2 Each TCLP method blank is identified in the TCLP extraction logbooks by a seven-character code. The first three characters are “PBT”, which stands for “Preparation Blank - TCLP”. Characters 4 through 6 consist of the three-digit preparation number of the extraction fluid. The seventh character is a letter, starting with “A” and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, “PBT316A” refers to the first TCLP method blank extracted using fluid from Batch 316; “PBT316B” refers to the second TCLP method blank extracted using the same fluid. The extraction date of each TCLP method blank is recorded in the TCLP Fluid Preparation and Use Logbook. For every TCLP method blank prepared, at least one matrix-spiked aliquot must be prepared with a sample associated with that TCLP method blank. Record the sample chosen and spiking amounts in the Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook. See Section 8.2 for matrix spiking procedure.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste

property exceeds the regulatory level. Because the laboratory charges for the preparation and analysis of TCLP matrix spikes, selection of samples for TCLP matrix spiking is left to the discretion of the client. A minimum of one TCLP matrix spike must be analyzed for each batch of 20 TCLP extractions. As a minimum, follow the matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 4 and Figures 4 and 5.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before any preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 Instructions for preparing TCLP matrix spikes for metals analysis are contained in Table 4. Instructions for preparing TCLP matrix spikes for organics analyses are contained in Figures 4 and 5. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\text{Recovery (\%)} = 100\ (X_s - X_u)/K$$

where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the TCLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will

require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall be followed.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 1311

Federal Register, Volume 55, Number 126, Friday, June 29, 1990, PP 26986-26998

Federal Register, Volume 57, Number 227, Tuesday, November 24, 1992, PP 55114-55117

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

List of Tables and Figures

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical methods.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 TCLP extractions performed (required). One per TCLP method blank (required). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within ± 20% of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-510-11	EPA METHOD 1311
Reagents	Extraction Fluid #1 prepared using sodium hydroxide pellets. 5% HNO3	Extraction Fluid #1 prepared using 1N sodium hydroxide solution. 1N HNO3
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike **recommended** for each waste type.	Matrix spike **required** for each waste type.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresol	200.0
m-Cresol	200.0
p-Cresol	200.0
Cresol	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
2,4-Dinitrotoluene	0.13
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13
Hexachloro-1,3-butadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6- Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl Chloride	0.2

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 4

TCLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 50 mL Final Volume (mL)**
TCLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
TCLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.10

Note: Spiking must be performed after TCLP extraction and before preservation.

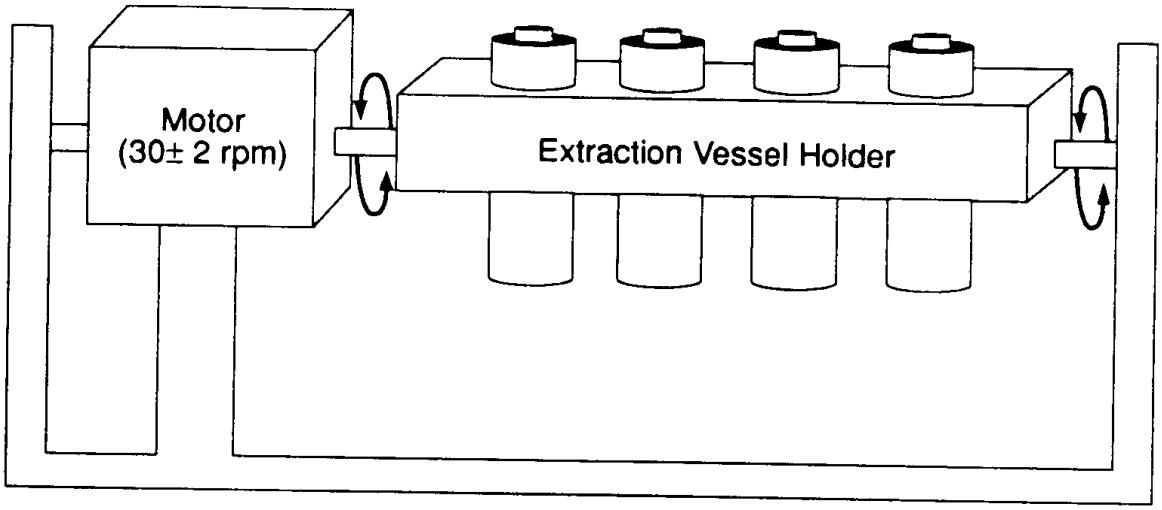
PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	**CONCENTRATION IN SOLUTION, mg/L**			
	TCLP Matrix Spike	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**1000 ug/L Hg Std.**
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 1

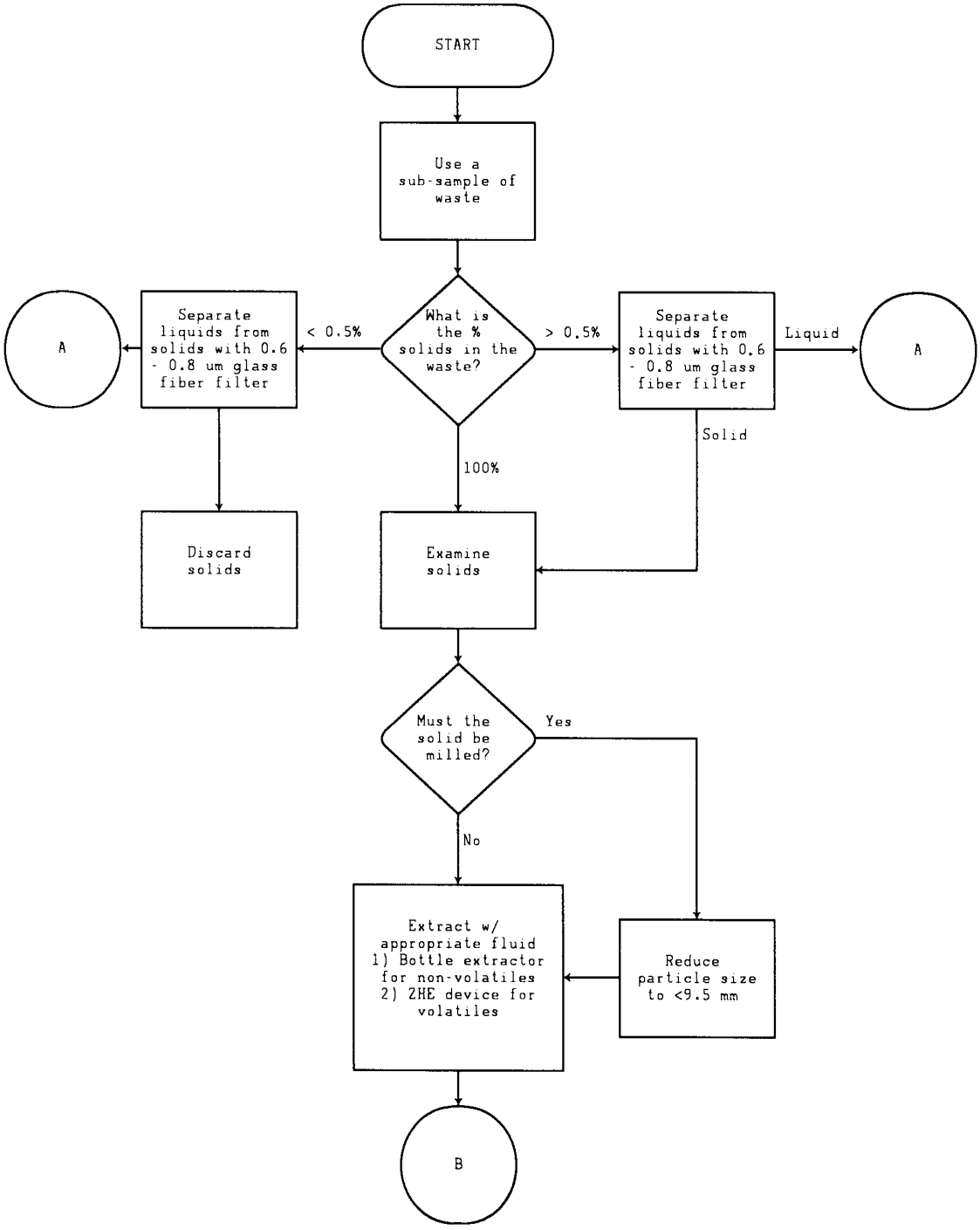
ROTARY AGITATION APPARATUS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 2

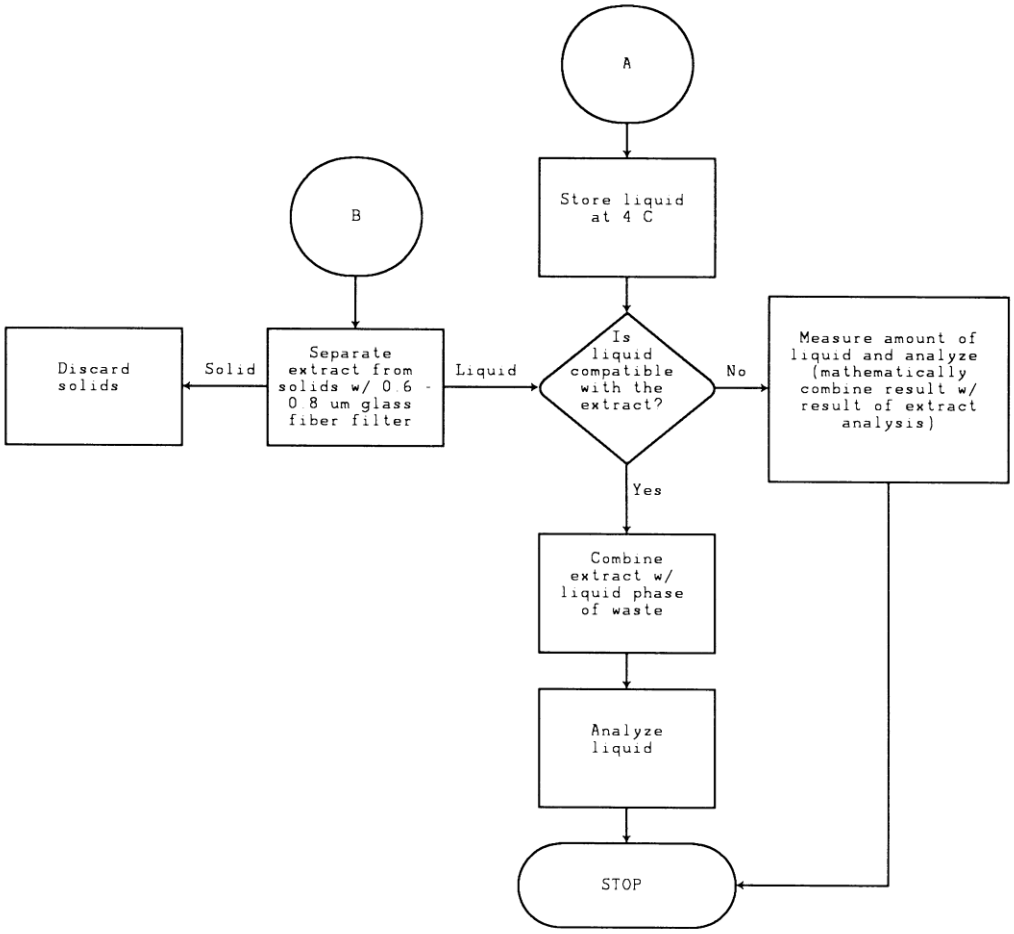
TCLP FLOW CHARTS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 3

TCLP FLOW CHARTS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 4

SVOA TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorphenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

* Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for TCLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 5

PESTICIDE TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)
Tetrachloro-m-xylene (TCMX)

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 6

EXAMPLE PAGE FROM TCLP FLUID USE LOGBOOK

Katahdin Analytical Services, LLC

Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook

FLUID PREPARATION					
TCLP ☑ SPLP ☐	TCLP Fluid #:	Fluid Batch #:	Prep Date:	Prepared by:	Measured pH:
	#17 1	1412	8/24/17	AMJ	4.88

Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)
Glacial Acetic Acid	MSR42	120	N.A.	20
Sodium Hydroxide	MSR54	N.A.	54.23	↓
0.6% Sulfuric Acid / 0.4% nitric acid				

8/24/17 AMJ

Matrix Spike: 2.0mL MW17211/MW17190 + 0.2mL MS2040/MW17209

FLUID USE LOG					
Katahdin Sample Number	TCLP Extraction Start Date	Extract to be Analyzed for:			
		Metals	SVOA	Pest	Herb
Blank: PBT1412A	8/24/17	✓	✓	✓	✓
Matrix Spike: SL7576-1B		✓	✓	✓	✓
SL7576-1B		✓	✓		
SL7576-2B		✓	✓		
SL7576-3B		✓	✓		
SL7576-4B		✓	✓		
SL7576-5B		✓	✓		
SL7699-1D	8/29/17	✓	✓	✓	✓
SL7794-4A	9-5-17	✓			
SL7861-1C		✓	✓	✓	✓
SL7861-2C		✓	✓	✓	✓

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AMJ 9/6/17

QAAA185

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TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 7

EXAMPLE PAGE FROM NON-VOLATILE TCLP EXTRACTION LOGBOOK (Page 1)

KATAHDIN ANALYTICAL SERVICES, LLC. **Non-Volatile TCLP/SPLP Extraction Log**

I. EXTRACTION CONDITIONS

Extraction Method:	SW846 1311 (TCLP) ☑		Balance ID: BAL-15	Rotary Extractor ID: 2		
	SW846 1312 (SPLP) ☐		pH Meter ID: Orion 520A s/n 7422	pH Probe ID: 5933576-0038		
Solid pH Determination:	Date: 11/29/19	Analyst: AB	Room Thermometer ID: Dig 70	(Room Temp Criteria. 23(±2)°C)		
Rotary Extraction Started:	Date: 11/29/19	Time: 1224	Analyst: AB	RoomTemp(°C):	Start: 22.3	End: 23.8
Rotary Extraction Completed:	Date: 11/30/19	Time: 0625	Analyst: AB	Room Temp (°C):	Min. 22.3	Max. 23.9
Extraction Filtered:	Date: 11/30/19	Time: 731	Analyst: AB	Filter Lot #: R8HA81937		
Elapsed Extraction Time (HH:MM): 18:01		5% HNO_3 ID (used to wash filters): MR2238		HNO_3 Lot # (used to preserve extracts): MSR130		
Fluid 1 pH (Day of use): 4.97		Fluid 1 Expiration Date: 11/28/20		Rotary Extractor Rotation Rate Checked? ✓ (30)		
Fluid 2 pH (Day of use):		Fluid 2 Expiration Date:		Criteria. 30 ± 2 RPM		

II. EXTRACTION SETUP

TCLP Fluid pH: #1 - 4.93 ± 0.05 #2 - 2.88 ± 0.05 SPLP Fluid pH: #1 - 4.20 ± 0.05 #2 - 5.00 ± 0.05

Katahdin Sample No. (include bottle ID)	Matrix	Check One: 100% Wet Solids- waste will yield no liquid upon filtration	Check One: < 100% Wet Solids (Perform Solids Determination below)	SPLP FLUID # (1 for east and 2 for west of Mississippi River)	TCLP pH Determination and Fluid Selection (date & init. above): Initial pH of solid phase: (If <5, use Fluid #1; if >5 add 3.5 mL of 1 N HCl)	TCLP pH Determination and Fluid Selection (date & init. above): pH after 1 N HCL addition (if <5, use Fluid #1; if >5, use Fluid #2	Extraction Setup: Volume of Extraction Fluid (mL)	Extraction Setup: Fluid # used	Extraction Setup: Associated Extraction Blank ID:	Extraction Setup: Weight of Waste (g)	pH of extract after extraction:	Extract to be analyzed for: Metals (M), SVOA (S), PEST (P), HERB (H), Cyanide (C)	Extraction Bottle ID (if applicable)
SM0864-1A	SL	✓	–	–	9.90	0.30	2000	1	PBT1505	100.07	6.36	M	N/A
-2A	↓	↓	–	–	9.93	0.26	↓	1	↓	100.08	6.76	↓	↓
-3A	↓	↓	–	–	9.46	0.12	↓	1	↓	100.04	4.98	↓	↓
-4A	↓	↓	–	–	7.12	0.17	↓	1	↓	100.03	5.00	↓	↓
												AB 11/30/19	

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QAAA195 0000070

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 8

EXAMPLE PAGE FROM NON-VOLATILE TCLP EXTRACTION LOGBOOK (Page 2)

III. SOLIDS DETERMINATION

Date AQ Filtered: 1/29/19

Katahdin Sample No. (include bottle ID)	AQ or SL	Time Filt'd	A Weight of filter (g)	B Weight of filtrate vessel (g)	C Weight of weigh boat + waste (g)	D Weight of weigh boat + residue (g)	E Weight of waste (C-D) (g)	F Weight of filtrate vessel + filtrate (g)	G Weight of liquid phase (F-B) (g)	H Percent wet solids [(E-G)/E x 100%]	I Weight of filter + dry solids (g)	J Weight of dry solids (I-A) (g)	K Percent dry solids (J/E x 100%)
SM0899-1K	AQ	1413	1.13	13.94	126.03	13.55	112.48	129.01	115.07	2.30	1.47	0.34	0.30

AB 1/30/19

IV. PHASE SEPARATION

Katahdin Sample No. (include bottle ID)	Matrix	Percent dry solids [1] <0.5%	Percent dry solids [1] >0.5%	L Weight of filtrate vessel (g)	M Weight of weigh boat + waste (g)	N Weight of weigh boat + residue (g)	O Weight of waste (M-N) (g)	P Weight of filtrate vessel + filtrate (g)	Q Weight of liquid phase (P-L) (g)	R Volume of liquid phase (mL) [2]	S Weight of wet solids (O - Q) (g)

AB 1/30/19

1) If dry solids is <0.5%, filter sufficient volume of waste to support all required analyses. If dry solids >0.5% and wet solids <100%, perform phase separation (steps L – S above).
2) If miscible, proportionately combine pre-extraction filtrate with rotary extract. If not miscible, analyze aliquots separately and mathematically combine results.

V. COMMENTS:

Reviewed By _____ Date 1-30-19

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TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

Prepared By: Mike Thomas Date: 4/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 2/1/01

Operations Manager: Joel C. Beutel Date: 1/30/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis F. Hughes Date: 2/1/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention. Changed from tabular to paragraph format (procedure section).	DN	1.31.01	1/31/01
02	Minor changes in sections 4, 5 and 7. Changes to Table 3.	DN	4.2.02	4.2.02
03	Changed HPLC to SPE. Updated extract methods, procedure and fractionation to concur with Method. Updated Tables 1 & 2, and Figures 1 & 2. Removed (old) Figures 2 & 3.	LAD	051305	05.305
04	Added variances between SOP and method updated Fractionation procedure. updated logbook	LAD	04/06	04/06
05	Updated solvent exchange procedure. Added fractionation solution and fractionation surrogate to Sect. 5. Added cartridge interference. Added waste disposal. Updated method reference.	LAD	09/07	09/07

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Changed the addition of fractionation surrogate from after KD process to immediately before fractionation, weights from 0.1 to 0.05 g, soxhlet extraction time from 16 – 24 to 18 ± 2, addition of hexane from 18 to 17 mL. Added the final volume in LB should be 2 mL, and to record hexane and methylene chloride volumes used in LB. Added KAS SOP CA-108 reference for additional subsampling information.	LAD	03/09	03/09
07	Added fractionation by HPLC to all applicable Sections. Added HPLC Maintenance Logbook and Run log examples. Added HPLC instrument parameters. Change surrogate Chloro-octadecane to 5-alpha androstane. Added references.	LAD	04/10	04/10
08	Section 4 - Added equipment and materials for microwave digestion. Section 7 - Added microwave digestion, added weekly fractionation checks, minor changes (donct decant, minimum of 10g I.V., 5mL/min HPLC flow rate, using reference bottle for I.V. determination). Added references to Section 10.	LAD	02/12	02/12
09	Sect. 7.47 - updated Fractionation Check Prep. Sects 4.23, 4.24, 7.49 → 7.58, Tables 5 and 6 - updated for new HPLC for fractionating. Figures 1, 2, 3 and 4 - Updated	LAD	05/13	05/13
10	Sect. 7 - Changed "Shake" time from 3 to 6 minutes. Added hexane is added to the KD throughout Na$_2$SO$_4$ funnel right after sample; 15-20 mL of hexane is used. Added wording about decanting samples. Changed KAS INC to KAS throughout.	LAD	08/15	08/15
11	Sect. 7 - Significant changes throughout entire section for clarity and to accurately reflect current practices. Sect 5 ~~error~~ updated materials. Sect 6 - updated reagents/Standards Updated Table 3, Figure 1 and Figure 2	LAD	12/17	12/17
12	Sect. 7 - Changed initial volume determination to marking meniscus w/ grease pencil, Changed methylene chloride extraction volume to 60mL Updated Logbook example	LAD	01/18	01/18
13	Sect. 7 - Clarified amount of DI added to the microwave sample insert. Updated references.	LAD	07/19	07/19

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-511-13**, titled **EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP-EPH METHODS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-511-13**, titled **EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP-EPH METHODS.**

Recipient: _____ Date: _____

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

1.0 SCOPE AND APPLICATION

This document describes the protocols of the Massachusetts Department of Environmental Protection Method used for the Determination of Extractable Petroleum Hydrocarbons (EPH), including sample collection, sample extraction and cleanup. The MADEP EPH method is designed to measure the collective concentrations of extractable aliphatic hydrocarbons within the ranges C_9 through C_{18} and C_{19} through C_{36}. This method is also designed to measure the collective aromatic hydrocarbons within the C_{11} through C_{22} range and to measure the individual concentrations of targeted polynuclear aromatic hydrocarbons (PAHs) in water and soil. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150°C and 265°C. Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum napthas, and other petroleum products which contain a significant percentage of hydrocarbons lighter than C_9 or for petroleum products which contain a significant percentage of hydrocarbons heavier than C_{36}.

1.1 Definitions

Extractable Petroleum Hydrocarbons (EPH) - all hydrocarbon compounds eluting from n-nonane to n-hextriacontane, excluding Targeted PAH Analytes. EPH is comprised of C_9 through C_{18} Aliphatic Hydrocarbons, C_{19} through C_{36} Aliphatic Hydrocarbons, and C_{11} through C_{22} Aromatic Hydrocarbons. EPH concentration data are reported as a toxicologically-weighted summation of the aliphatic and aromatic hydrocarbon fractions.

C_{11} through C_{22} Aromatic Hydrocarbons - all aromatic hydrocarbon compounds eluting from naphthalene through benzo(g,h,i)perylene, excluding Targeted PAH Analytes.

C_9 through C_{18} Aliphatic Hydrocarbons - all aliphatic hydrocarbon compounds eluting from n-nonane to just before n-nonadecane (n-C_{19}).

C_{19} through C_{36} Aliphatic Hydrocarbons - all aliphatic hydrocarbon compounds eluting from n-nonadecane through n-hexatriacontane (n-C_{36}).

Targeted PAH Analytes - the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 3.

1.2 Responsibilities

Implementation of this SOP requires sufficiently trained analysts and properly functioning instrumentation. Samples must be properly extracted following Katahdin

Analytical Quality Assurance/Quality Control requirements. Only analysts/technicians qualified and experienced with this method may perform these procedures. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in the determination of Extractable Petroleum Hydrocarbons to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for EPH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

All standards should be prepared in a hood.

1.4 Waste Disposal and Pollution Control

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated (during the rinsing of glassware, etc.) should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, as well as cartridges used for the fractionation of EPH samples, should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

A soil or water sample is extracted with methylene chloride, dried with sodium sulfate, solvent exchanged into hexane, and then concentrated using a Kuderna-Danish apparatus. Sample fractionation into aliphatic and aromatic fractions is conducted using a preparative HPLC column and Foxy fraction collector. The two extracts produced are then re-concentrated to final volumes of 1 mL each. The resulting extracts (an aliphatic extract and an aromatic extract) are analyzed separately for EPH using a GC equipped with a flame ionization detector (FID).

3.0 INTERFERENCES

Method interferences are minimized by using high purity reagents and by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone and methylene chloride.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interference will vary from one source to another. A preparative HPLC column cleanup and fractionation procedure is used to overcome many of these interferences, but some samples may require additional cleanup approaches that are beyond the scope of this method.

Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable Petroleum Hydrocarbons. If necessary or desirable, additional sample cleanup or analytical procedures may be employed to minimize or document the presence of such compounds.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

The leaching of plasticizers and other compounds has been observed from commercially available silica gel cartridges used to fractionate EPH sample extracts. Concerns of this nature must be continuously monitored and documented by the analysis of laboratory method blanks.

4.0 APPARATUS AND MATERIALS

4.1 1-L amber glass bottles

4.2 4 oz. (120 mL) glass wide-mouth jars

4.3 Vials: 4 mL glass vials with silicone/PTFE septa in open top caps

4.4 Vials: 1.8 mL with silicone/PTFE septa in open-top caps

4.5 Glass funnels

4.6 2-L Separatory teflon SEP funnels with screw closures

4.7 Kuderna-Danish (K-D) apparatus including 10-mL concentrator tube, 500-mL Evaporative flask and 3-ball Snyder column

4.8 12 mL conical vials

4.9 250 mL amber bottles with Teflon-lined screw covers

4.10 Disposable pipettes: Pasteur, 5 ¾”

4.11 25-mL graduated cylinder

4.12 1-L graduated cylinder

4.13 400-mL beakers

4.14 A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples

4.15 Nitrogen blowdown apparatus: Organomation N-EVAP

4.16 Water bath: heated with a concentric ring cover, capable of temperature control ($\pm$2° C). The bath should be used in a hood

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

4.17 Syringes, gas tight, 1.0mL, Hamilton or equivalent, and 10mL Luer-Lock syringes (Popper or equivalent)

4.18 Syringe filters, 13 mm diameter

4.19 Boiling Chips, silicon carbide (carborundum), 12 mesh

4.20 Soxhlet extraction apparatus, or apparatus for equivalent technique such as ASE (accelerated solvent extractor)

4.21 VOA vials, 40 mL

4.22 Filter paper, 18.5 cm diameter

4.23 Wide range pH test strips, CF-type, pH0-14

4.24 Solid Phase Extraction (SPE) cartridges with silica gel (5g/20mL)

4.25 HP Series 1100 HPLC Quaterrnary pump, autosampler, or equivalent.

4.26 Foxy R2 fraction collector, or equivalent

4.27 Restek HPLC column, or equivalent, 150mm X 10mm.

4.28 Milestone Ethos Ex microwave

4.29 PRO-24 rotor with top

4.30 TFM sample inserts and caps

4.31 Microwave pressure vessels and covers

4.32 Fiber optic Thermowell

4.33 Stirbars

4.34 Black wafers for polarizing

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water: organic free water

5.2 Solvents: hexane, methylene chloride, and acetone; pesticide grade or better

5.3 Sodium sulfate: (ACS) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray (sodium sulfate may be purchased pre-purified by the manufacturer)

5.4 Sand: free of extractable petroleum hydrocarbons, purified by heating at 400 □C for four hours

5.5 Matrix Spike/Lab Control Sample Spiking Solution: 31 compounds (See Table 3)

5.6 Surrogate Spiking Solution: 5-alpha androstane and ortho-terphenyl (OTP) at concentrations of 90 ug/mL in acetone

5.7 Fractionation Check Standard: 31 compounds and 5-alpha androstane and ortho-terphenyl (OTP) (See Table 3)

5.8 Fractionation Surrogate Solution: 2-Fluorobiphenyl and 2-Bromonaphthalene at 90 ug/mL in hexane

5.9 Hydrochloric acid solution (1:1 HCl: H_2O) - slowly add 500 mL of HCl to 500 mL reagent water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps.

6.2 Soil and sediment samples are collected in 4 oz (120 mL) wide-mouth glass jars with Teflon-lined screw caps.

6.3 Sample container, preservation and holding times are summarized in Table 4.

6.4 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the date and time of sampling and any preservatives added.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (if applicable):

- Extraction and Analytical methods
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper, vials
- Nitrogen evaporation water bath temperature(s)

- Sonicator Amplitude
- Sample pH (if applicable)
- Extraction and Concentration dates
- Extraction and Concentration analysts
- Sample ID or QC sample ID
- Initial and final volumes or weights
- Surrogate and spike amounts
- Boiling chip date
- Balance ID
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- Lot numbers of the vials the concentrated extracts are stored in.
- Soxhlet or microwave start/end date and times

Samples are extracted using methylene chloride, and solvent-exchanged into hexane. The recommended extraction procedure for water samples is a separatory funnel liquid-liquid extraction technique based upon SW-846 Method 3510C. Soil or sediment samples are extracted using either Soxhlet extraction method SW-846 3540C or by microwave method SW-846 3546. Alternative extraction procedures are acceptable such as SW-846 3541 automated Soxhlet extraction procedure, provided that the laboratory can document acceptable performance. Sonication (SW-846 3550C) may only be used for the extraction of highly contaminated (free product) non-soil/sediments.

AQUEOUS EXTRACTION

7.1 With a grease pencil, mark the sample bottles at the water meniscus for later determination of sample volume. Transfer the entire contents of each sample to a 2-liter separatory funnel.

7.2 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, pour 1 liter of laboratory reagent grade water into a 2-liter separatory funnel.

7.3 A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS/LCSD, pour 1-liter of laboratory reagent grade water into each of two 2-liter separatory funnels.

7.4 A matrix spike/matrix spike duplicate (MS/MSD) is prepared per client request. With a grease pencil, mark the sample bottles at the water meniscus for later determination of sample volume. Transfer each to a 2-liter separatory funnel.

7.5 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of EPH surrogate spiking solution (section 5.6) using a pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.6 To LCS/LCSD and MS/MSD add 1.0 mL of the EPH matrix spike/LCS spiking solution (section 5.5) using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.7 Check the pH of the sample(s) with wide-range pH paper. If the pH of a sample or MS/MSD is >2, acidify to a pH <2 by adding 1:1 HCl. Each method blank and LCS/LCSD must also be adjusted to pH <2 by adding 1:1 HCl. Adjust all samples and quality control samples as needed with 1:1 HCl. Record any unpreserved samples and/or MS/MSD's (initial pH >2) in laboratory notebook.

7.8 Add 60 mL methylene chloride to the sample bottles, blank bottle and LCS/D bottles to rinse the inner walls of the container, then add this solvent to the separatory funnel.

7.9 Seal, shake and vent the separatory funnel. Place separatory funnel on an automatic shaker and shake vigorously for 6 minutes.

NOTE: Methylene chloride creates excessive pressure very rapidly.

7.10 Allow the organic layer to separate from the water phase. If the emulsion interface between layers is more then one-third the size of the solvent layer, the analyst/technician must employ mechanical techniques to complete the phase separation and note the techniques in the laboratory notebook. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in a 250 mL amber bottle with a Teflon-lined screw-cap.

7.11 Repeat the extraction two more times using additional 60 mL portions of solvent. Collect the methylene chloride layer in the same amber collection bottle (Steps 7.8 to 7.10).

7.12 Fill the sample bottle with reagent water to the grease pencil marking. Measure the volume of reagent water using a graduated cylinder. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.13 See sections 7.40 through 7.47 for the concentration of the extracts.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

SOIL EXTRACTION – SOXHLET

7.14 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.15 Add ~ 250 mL of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.16 Samples should be prepped as follows:

7.16.1 Do not decant or discard any water layer on a sediment sample.

Note: Some work orders may specify to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.16.2 Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook.

7.16.3 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate (see step 7.16) to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.16.4 Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“, for more detailed guidance on sub-sampling to ensure reproducibility.

7.17 **The following steps should be performed rapidly to avoid loss of the more volatile extractables.** Weigh out 10.0 ± 0.05 g or greater portion of sample into a labeled 400 mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 10 g and 20 g of anhydrous powdered sodium

sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (i.e. low moisture content will require less sodium sulfate, and vice-versa). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

Note: If transferring samples/QC samples to Soxhlet extractors (step 7.19) immediately after weighing out, covering the beakers with foil is not necessary.

7.18 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 10.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 20 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 20 g sodium sulfate, the method blank must contain 20 g in order to evaluate the sodium sulfate as a potential source of contamination. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.19 A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS/LCSD, weigh out two 10.0 ± 0.05 g portions of purified sand in labeled 400 mL beakers. Add 10 g sodium sulfate to each and mix well. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.20 To prepare MS/MSD, weigh out 10 ± 0.05g or greater portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add between 10 g and 20 g sodium sulfate to each to produce a free-flowing mixture, and mix well. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.21 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.22 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of EPH surrogate spiking solution (section 5.6) using a pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.23 To LCS/LCSD and MS/MSD add 1.0 mL of the EPH matrix spike/LCS spiking solution (section 5.5) using a 1.0 mL gas tight syringe. Record matrix spike/LCS

spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.24 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the flat-bottom flasks when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 45-50% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until reaching the level of the small siphon tube, then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 20 ± 2 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.25 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.26 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container ("I" waste). It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

7.27 See sections 7.49 through 7.47 for the concentration of the extracts.

SOIL EXTRACTION – MICROWAVE

7.28 Prepare samples as in section 7.15.

7.29 Rinse all glassware, TFM sample inserts, the thermowell, and stirbar three times with methylene chloride. Press all TFM sample insert caps with a re-conditioning tool to ensure a snug fit into inserts. Rinse insert caps with methylene chloride.

7.30 Add a stir bar into all TFM sample inserts.

7.31 A method blank and associated LCS/LCSD must be prepared with each extraction batch, not to exceed 20 samples. Weigh 10.0 g ± 0.05 g of purified sand into

sample insert and add 2 mL DI water if no wafer is used. Add a black wafer or 2 mL of DI water to the method blank and LCS/LCSD to increase polarity.

7.32 **The following steps should be performed rapidly to avoid loss of the more volatile extractables.** For each sample and MS/MSD, weigh out a 10.0 ± 0.05 g or greater portion into a labeled TFM insert. Record the weight to the nearest 0.01 g in appropriate extraction logbook.

7.33 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of EPH surrogate spiking solution using a pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.34 To LCS/LCSD and MS/MSD add 1.0 mL of the EPH matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.35 To all samples and QC, add approximately 25 mL of methylene chloride, ensuring that all of the sample is completely saturated. To avoid overpressurizing the sample, do not fill the insert more than ¾ of the way (including the sample and methylene chloride). Put sample insert caps on.

7.36 Chose the wettest sample to be the reference sample and put the fiber optic thermowell through the top. **Be sure to only run samples with close to the same % moisture. Samples and QC may need to be extracted in separate microwave runs depending on the moisture.**

7.37 Put samples in the microwave vessels and tighten the top until flush. The reference sample has a different cover with a loose piece of teflon; make sure that it does not get lost. Place the vessels in the rotor, noting which samples go into which spot. Place the rotor top on. Carefully insert the fiber optic probe into the reference vessel. Put the rotor in the microwave and plug the fiber optic in. Close the door.

7.38 Using the microwave software, test the rotor to ensure the fiber optic won't bind during the run. Select the appropriate method. Hit start, test the stirring mechanism, and hit start again. The extraction program takes 40 minutes, including a 20 minute temperature ramp and a 20 minute hold at a temperature of 115 ^{o}C, as recommended in method SW-846 3546. Allow the samples to cool for 20 minutes. Once the reference sample has reached a temperature that is below the boiling point of the solvent(s) used, the samples are safe to open. **Be sure to remove the rotor from the microwave before the fiber optic is removed from the reference sample.**

7.39 See sections 7.40 through 7.47 for the concentration of the extracts.

CONCENTRATION OF EXTRACTS

7.40 Rinse the K-D glassware (flask, concentration tube, and three ball Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels.

7.40.1 **Note**: The lot number of the filter paper must be recorded in the extraction logbook.

7.41 Pour the extracted samples from their containers (the containers should be either 250 mL amber bottles, soxhlet extract flasks, or microwave sample inserts, depending on the extraction method) directly into the KD apparatus through the sodium sulfate in the funnels. Transfer the labels from the extract flasks to the K-D flasks as you transfer the samples. After pouring all of the extract volume through the sodium sulfate, rinse the containers three times with ~ 3 mL of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mL of methylene chloride and allow to drain.

7.42 Add approximately 15-20 mL of Hexane through the sodium sulfate filled funnel and into the KD body to allow for a solvent exchange. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only.

7.43 Place the K-D apparatus on a hot water bath set to 75^{o}C. Gently swirl K-D in water until boiling begins. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume of liquid reaches 4 - 6 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Rinse the Snyder column lower joint with ~ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ~1 mL hexane.

7.44 Reduce the hexane extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45^{o}C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipette must be rinsed down at least once or twice with ~1 mL of hexane. The solvent level in the

concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.45 Bring extract to 1 mL (add hexane or blow down more if needed) and place in a 4.0 mL vial. Record the "pre-fractionation" final volume as 1 mL in the extractions logbook.

7.46 Record the sample preparation information for the extraction and concentration steps. See list at beginning of section 7.

7.47 The 1 mL extract is now ready to be cleaned and fractionated using HPLC or SPE fractionation. Proceed to step 7.47.

FRACTIONATION

7.48 Fractionation can be performed either by High Performance Liquid Chromatography (HPLC) or through Solid Phase Extraction (SPE). HPLC is the method typically used at Katahdin Analytical Services. The SPE cartridge method can be performed in the event that the HPLC is unavailable.

7.49 Prior to fractionation by either method, a Fractionation Check is made by preparing a known volume of the Fractionation Check Standard (Table 3) and mixing it with an equal volume of Fractionation Surrogate Solution (Section 5.8). The mixture is the Fractionation Check.

Note: The Fractionation Check includes the EPH spike compounds, EPH surrogate compounds, and fractionation surrogate compounds.

7.50 The Fractionation Check is labeled with the creation lot number and date of fractionation. Prior to sample fractionation, run the fractionation check on the fractionator, then analyze it in order to confirm the amount of hexane necessary to prevent aromatic breakthrough into the aliphatic fraction and to ensure the fractionator is working properly and does not need service or maintenance.

7.50.1 For HPLC fractionation: A Fractionation Check is analyzed by FID on a weekly basis before any samples are fractionated.

7.50.2 For SPE fractionation: A Fractionation Check is run with each new lot of SPE cartridges or every three months.

FRACTIONATION – HPLC

7.51 Add 1 mL of fractionation surrogate to each 1 mL extract in the 4 mL vials (see step 7.46).

7.52 Filter samples using 10 mL Luer-Lok syringes and 13 mm syringe filters prior to fractionation and collect filtered samples in 1.8 mL vials. Cap the vials.

7.53 Make sure the methylene chloride and hexane bottles are full. All solvents need to be sonicated for at least 15 minutes prior to being added to the HPLC03 instrument.

7.54 At the controller, make sure the flow is set to 1.00 mL/min, then turn the system on. This starts the flow. The system automatically sets solvent A (hexane) at 100%.

7.55 Prime the pumps for line A.

7.56 Set solvent D (methylene chloride) to 100%.

7.57 Prime the pumps for line D.

7.58 When finished priming, change pump back to 100% hexane by entering 0 for solvent D, the system will automatically switch solvent back to hexane.

7.59 Turn on the Isco Foxy 200 fraction collector using the power button.

7.60 Load the Foxy tray with 40 mL VOA vials. You will need two vials per sample, the first being for the aliphatic and the second for the aromatic. Appropriately label each VOA vial.

7.61 After the vials are on the tray push the “play” button on the front of the Foxy. The sampling arm should move to the ready position waiting for the injection to occur.

7.62 Load tray with samples to be injected in order from lightest to darkest samples. QC is usually placed in front after a rinse. Make sure to add extra rinses at the end when working with dark samples. On the controller, change the starting vial and ending vial at the top of the screen. Once the sample tray and Foxy tray is set up, you may start the system to begin collecting. Highlight and select the start system button. Most injections are 1.0 mL, however you can inject a different volume as long as you make sure there is enough sample in the vial to inject (try to have at least 200 uL extra volume). When the injection occurs the sampling arm of the Foxy will position itself over the aliphatic vial and start collecting. When the aliphatic

portion finishes the arm automatically moves to the second vial and immediately starts collecting the aromatic fraction.

7.63 The flow time for hexane is 4 min, while methylene chloride is 9 min. The collect time for hexane is 4.25 min, while the methylene chloride is 7.00 min.

7.64 Place all fractions on the N-evap once the fractionation is complete. The fractions should be left in the VOA vial until each reaches a suitable volume to transfer into a 12 mL conical vial with graduated volume markings.

7.65 All fractions in the 12 mL conical vials should be placed on the N-EVAP bath and blown down (see section 7.41) to the volume at which they were injected. (If 1.0 mL was injected, the final volume will be 1 mL, if 0.5 mL was injected the final volume will be 0.5 mL.) The final volume recorded in the logbook and computer, however, should be 2.0 mL (for a 1 mL injection) since this is what the actual final volume is. All aliphatic fractions are in Hexane, while all aromatic fractions are in methylene chloride.

7.66 Transfer all fractionated samples to 1.8 mL screw top vials (one for each fraction).

7.67 Samples are now ready for analysis.

7.68 All samples fractionated on the HPLC should be written in the HPLC03 logbook (figure 3).

FRACTIONATION - SPE CARTRIDGE

NOTE: If using the SPE cartridges: The Fractionation step is a critical yet highly sensitive procedure. Small changes in the volumes of eluting solvents, fractionation equipment, and/or fractionation techniques can significantly impact the proportion of hydrocarbons segregated in either the aliphatic or aromatic fractions. Considerable care and attention is required to ensure satisfactory results.

7.69 Place SPE cartridges on the sample collection apparatus. With the valve under the sample in the open position, rinse each SPE cartridge with 30mL of hexane, using a 10mL syringe. Close the valve when approximately 0.5 cm of solvent remains above the cartridge frit. Do not allow the silica gel cartridges to dry out after the solvent has passed through. Empty the collection apparatus of eluted solvent.

7.70 Place a 40mL VOA vial, labeled as the aliphatic fraction, under each SPE cartridge in the sample collection apparatus. Add 1mL of the fractionation check or sample to the top of the SPE cartridge. Open the valve. Just as the sample/solvent layer reaches the frit, add ~17mL of hexane by syringe. (The amount of hexane is determined by the fractionation check done on each lot or every three months.)

The solvent should be added slowly, however, the cartridge must not be allowed to go dry. Rinsing the sides of the cartridge when adding the hexane may help improve recoveries. Close the valve when ately 0.5 cm of solvent remains. Remove the test tubes.

7.71 Place a new 40mL VOA vial, labeled as the aromatic fraction, under the sample collection apparatus. Open the valve. Just as the hexane layer reaches the frit, add 20mL of methylene chloride, by syringe, to collect the aromatic fraction. Rinsing the sides of the cartridge when adding the methylene chloride may help improve recoveries. Remove the test tubes.

7.72 All fractions should be placed on the N-EVAP bath and blown down (see section 7.41) to the volume at which they were fractionated (if 1.0mL was fractionated, the final volume will be 1mL). The final volume recorded in the logbook and computer, however, should be 2.0 mL since this is what the actual final volume is. All aliphatic fractions are in hexane, while all aromatic fractions are in methylene chloride.

7.73 Record the sample preparation information for the fractionation and concentration steps. At a minimum, record the SPE cartridges lot#, fractionation date, fractionation initials, volumes of hexane and methylene chloride added for each fraction, concentration volume, concentration initials, and any deviations or problems associated with the fractionation of the samples.

7.74 Transfer all fractionated samples to 1.8 mL screw top vials (one for each fraction).

7.75 Samples are now ready for analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) is required for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix
- Each extraction method
- Every extraction batch of twenty or fewer samples

Sample specific matrix spikes (MS) and matrix spike duplicates (MSD) are extracted per client request or per project requirements.

Refer to Katahdin SOP CA-322, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH), current revision, for other Quality Control parameters and acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to Katahdin SOP CA-322, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH), current revision.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004, revision 1.1.

Quality Control Requirements and Performance Standards for the *Analysis of Extractable Petroleum Hydrocarbons (EPH)* in Support of Response Actions under the Massachusetts Contingency Plan (MCP), July 1, 2010

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.01.1, 2018

Katahdin Analytical Services, Inc. SOP CA-322, current revision, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH)

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

LIST OF TABLES AND FIGURES

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

TABLE 1

QC REQUIREMENTS

Parameter/ Method	**QC Check**	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**
Sample Prep for waters and soils for EPH determination	Method blank	One per prep batch or 20 samples whichever is more frequent.	Refer to analytical method.	Refer to analytical method.
	LCS/LCSD	One each per prep batch	Refer to analytical method.	Refer to analytical method.
	Matrix Spike/Matrix Spike Duplicate	Per client request	Refer to analytical method.	Refer to analytical method.
	Sample Duplicate/ Matrix Spike	Per client request	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst performing the method, then yearly	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL and-or LOD/LOQ verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-511-13	MADEP METHOD, current revision
Apparatus/Materials	Automated HPLC and Fraction Collector	SPE cartridges
Reagents		
Sample preservation/ handling		
Procedures	1) Addition of 15-20 mL of hexane to KD body after transfer of extract ito KD apparatus to accomplish solvent exchange. 2) …with 20 to 30 mL of methylene chloride …to complete the quantitative transfer. 3) ... Place the K-D apparatus on a hot water bath (75^{o}C). Gently swirl K-D in water until boiling begins. 4) Fractionate samples using HPLC or SPE cartriges.	1) Addition of 50 mL of hexane to KD body after concentration of extract in methylene chloride to 1mL to accomplish solvent exchange. 2) 9.1.2.5 …with 100 to 125 mL of methylene chloride to complete the quantitative transfer. 3) 9.1.10 Place the K-D apparatus on a hot water bath (80-90 ^{o}C) so that… 4) Fractionate samples with SPE cartridges.
QC - Spikes	1) Surrogate, spike and fractionation surrogate all prepared at 90 ug/mL for use in a 2 mL or prefractionated volume. 2) Fractionation check solution: aliphatics, aromatics, 5-alpha androstane, and o-terphenyl at 200ug/mL each	1) Surrogate at 40 ug/mL, matrix spike at 50-150 ug/mL, fractionation surrogate at 40 ug/mL. 2) Fractionation check solution: aliphatics and aromatics at 200ug/mL each
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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TABLE 3

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, AND MATRIX SPIKE CONCENTRATIONS - MADEP - EPH METHOD

TYPE	ANALYTE	Stock Standards * (ng/uL)	Fractionation Check Standard ** (ng/uL)	Matrix Spike Standard *** (ng/uL)
Aromatic	Naphthalene	1,000	90	90
Aromatic	2-Methylnaphthalene	1,000	90	90
Aromatic	Acenaphthylene	1,000	90	90
Aromatic	Acenaphthene	1,000	90	90
Aromatic	Fluorene	1,000	90	90
Aromatic	Phenanthrene	1,000	90	90
Aromatic	Anthracene	1,000	90	90
Aromatic	Fluoranthene	1,000	90	90
Aromatic	Pyrene	1,000	90	90
Aromatic	Benzo(a)Anthracene	1,000	90	90
Aromatic	Chrysene	1,000	90	90
Aromatic	Benzo(b)Fluoranthene	1,000	90	90
Aromatic	Benzo(k)Fluoranthene	1,000	90	90
Aromatic	Benzo(a)Pyrene	1,000	90	90
Aromatic	Indeno(1,2,3-cd)Pyrene	1,000	90	90
Aromatic	Dibenzo(a,h)Anthracene	1,000	90	90
Aromatic	Benzo(g,h,i)Perylene	1,000	90	90
Aromatic	o-Terphenyl (surr)	1,000	90	90
Aliphatic	Nonane	1,000	90	90
Aliphatic	Decane	1,000	90	90
Aliphatic	Dodecane	1,000	90	90
Aliphatic	Tetradecane	1,000	90	90
Aliphatic	Hexadecane	1,000	90	90
Aliphatic	Octadecane	1,000	90	90
Aliphatic	Nonadecane	1,000	90	90
Aliphatic	Eicosane	1,000	90	90
Aliphatic	Docosane	1,000	90	90
Aliphatic	Tetracosane	1,000	90	90
Aliphatic	Hexacosane	1,000	90	90
Aliphatic	Octacosane	1,000	90	90
Aliphatic	Triacontane	1,000	90	90
Aliphatic	Hexatriacontane	1,000	90	90
Aliphatic	5-alpha androstane (surr)	1,000	90	90

* The Aromatic Hydrocarbon Stock Standards (17 PAH compounds and o-terphenyl) should be prepared in methylene chloride. The Aliphatic Hydrocarbon Stock Standards (consisting of 14 normal alkanes and 5-alpha androstane) should be prepared in hexane.

** The Fractionation Check Standard (31 compounds and two surrogates) should be prepared in hexane.

*** The Matrix Spike Solution should be prepared in acetone. The o-terphenyl and 5-alpha androstane surrogate solution should also be prepared in acetone.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

TABLE 4

HOLDING TIMES AND PRESERVATIVES FOR EPH SAMPLES

MATRIX	CONTAINER	PRESERVATION	HOLDING TIME
Aqueous Samples	1 Liter amber glass bottle with Teflon- lined screw cap	Add 5 mL of 1:1 HCl; cool to 4 (±2) °C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap	Cool to 4 (±2) °C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap. Jar should be filled to only 2/3 capacitiy to avoid breakage if expansion occurs during freezing.	Freeze at -10 °C in the field or in the laboratory*	Samples must be extracted within 14 days of the date thawed and extracts analyzed within 40 days of extraction*

* Samples processed in the laboratory must be preserved at 4 (±2) °C and frozen within 48 hours of the time of collection. Frozen samples may be held for up to one year prior to analysis and must be extracted within 24 hours of thawing.

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TABLE 5

HPLC PROGRAM 1 RUN PARAMETERS

RUN TIME (Min)	FLOW RATE (mL/min)	%A Hexane	%D $MeCl_2$
Initial	5	100	0
4	5	0	100
13	5	100	0
20	5	100	0

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

TABLE 6

ISCO FOXY R2 FRACTION COLLECTOR

- Use the time windows function
- Collect Vial 1 from 0.00 min – 4.25 min
- Collect Vial 2 from 4.25 min – 11.25 min
- Total fraction size is 11.25 min
- There is a 7 minute equilibration time in hexane where it diverts the hexane to waste until the new injection.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

FIGURE 1

EXAMPLE OF AQUEOUS EXTRACTION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, LLC.
ORGANIC EXTRACTIONS LOG - AQUEOUS FUEL OILS

Extraction Method: (check one)	SW846 3510 (SEP)			SW846 3520 (CLLE)			Nitrogen Water Bath Temperature:	
Analytical Method: (√ one)	SW8015M (DRO)	SW8100M (TPH)	MADEP EPH - Meniscus mthd must be used to determine IV.	ME DEP 4.1.25	FLO PRO	CT ETPH	TX 1005	Other:
Spike ID:		Surrogate ID:		Frac. Surrogate ID:			Other:	
MeCl2 Lot #:		Acetone Lot #:		n-Pentane Lot #:			Hexane Lot #::	
NaSO4 Lot #		Baked NaSO4 ID:		Filter Paper Lot # (KD)			Vial Lot #:	
Sample pH checked prior to extraction?		Yes	No	Note unpreserved samples in comments section.			Boiling Chip Date:	
Blank and LCSs adjusted to pH < 2?		Yes	No	pH Paper Lot #:			HCl Lot #	
Prep Start Time:		Prep End Time:		CLLE Start Time:			CLLE End Date & Time:	

Date Extracted	Ext. Init.	Sample ID	Init. Vol. (mL)	Surr Vol. (mL)	Spike Vol. (mL)	Final Vol Pre-Frac. (mL)	Date Conc. Pre-Frac.	Conc. Init. Pre-Frac.	Tray Location	Frac. Init.	Frac. Surr Vol. (mL)	Final Vol. Post-Frac. (mL)	Date Conc Post-Frac.	Tray Location	Conc. Init. Post-Frac.	Comments

EX-015 – Revision 6 – 10/03/2018

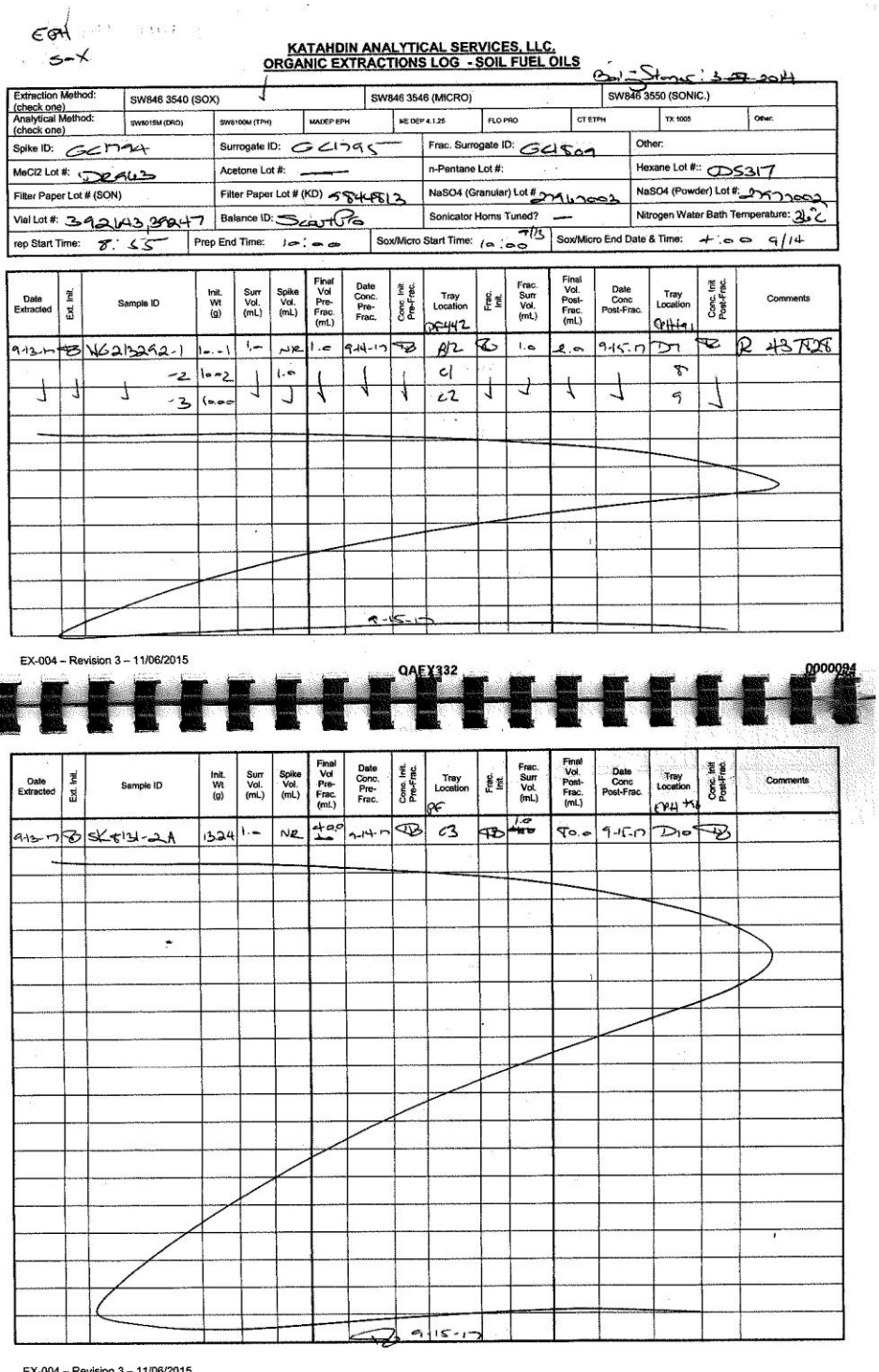
Date Extracted	Ext. Init.	Sample ID	Init. Vol. (mL)	Surr Vol. (mL)	Spike Vol. (mL)	Final Vol Pre-Frac. (mL)	Date Conc. Pre-Frac.	Conc. Init. Pre-Frac.	Tray Location	Frac. Init.	Frac. Surr Vol. (mL)	Final Vol. Post-Frac. (mL)	Date Conc Post-Frac.	Tray Location	Conc. Init. Post-Frac.	Comments

EX-015 – Revision 6 – 10/03/2018

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

FIGURE 2

EXAMPLE OF SOIL EXTRACTION LOGBOOK PAGE



EPH
S-X

KATAHDIN ANALYTICAL SERVICES, LLC.
ORGANIC EXTRACTIONS LOG - SOIL FUEL OILS

Bal = Stanley 3-5B-2014

Extraction Method: (check one)	SW846 3540 (SOX)		SW846 3546 (MICRO)		SW846 3550 (SONIC.)		
Analytical Method: (check one)	SW8015M (DRO)	SW8100M (TPH)	MADEP EPH	ME DEP 4.1.25	FLO PRO	CT ETPH	TX 1005	Other:
Spike ID: GC794	Surrogate ID: GC795		Frac. Surrogate ID: GC1509		Other:			
MeCl2 Lot #: DR963	Acetone Lot #: —		n-Pentane Lot #:		Hexane Lot #:: DS317			
Filter Paper Lot # (SON)	Filter Paper Lot # (KD) 584813		NaSO4 (Granular) Lot # 2711-2003		NaSO4 (Powder) Lot #: 2711-2003			
Vial Lot #: 392643 39247	Balance ID: Scientech		Sonicator Horns Tuned? —		Nitrogen Water Bath Temperature: 3.2°C			
rep Start Time: 8:55	Prep End Time: 10:00		Sox/Micro Start Time: 10:00 9/13		Sox/Micro End Date & Time: 10:00 9/14			

Date Extracted	Ext. Init.	Sample ID	Init. Wt (g)	Surr Vol. (mL)	Spike Vol. (mL)	Final Vol Pre-Frac. (mL)	Date Conc. Pre-Frac.	Conc. Init. Pre-Frac.	Tray Location	Frac. Init.	Frac. Surr Vol. (mL)	Final Vol. Post-Frac. (mL)	Date Conc Post-Frac.	Tray Location	Conc. Init Post-Frac.	Comments
9-13-17	TB	WG213292-1	10.-1	1.-	NR	1.0	9-14-17	TB	B2	TB	1.0	2.0	9-15-17	D7	TB	R 437428
↓	↓	↓ -2	10-2	↓	1.0	↓	↓	↓	C1	↓	↓	↓	↓	8		
↓	↓	↓ -3	10.00	↓	↓	↓	↓	↓	C2	↓	↓	↓	↓	9	↓	

9-15-17

EX-004 – Revision 3 – 11/06/2015

QAEX332

0000094

Date Extracted	Ext. Init.	Sample ID	Init. Wt (g)	Surr Vol. (mL)	Spike Vol. (mL)	Final Vol Pre-Frac. (mL)	Date Conc. Pre-Frac.	Conc. Init. Pre-Frac.	Tray Location	Frac. Init.	Frac. Surr Vol. (mL)	Final Vol. Post-Frac. (mL)	Date Conc Post-Frac.	Tray Location	Conc. Init Post-Frac.	Comments
9-13-17	TB	SK813-2A	1324	1.-	NR	400 1.0	9-14-17	TB	C3	TB	1.0	80.0	9-15-17	D10 EPH HC	TB	

9-15-17

EX-004 – Revision 3 – 11/06/2015

QAEX332

0000095

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

FIGURE 3

EXAMPLE OF HPLC03 LOGBOOK

Katahdin Analytical Services
HPLC03 Logbook

Analytical Method:	MA-EPH: ✓	Other:	Flow: 5.0mL/min
MeCl2 Lot #: DI071		Hexane Lot #: DH162	

Tray Pos. No.	Sample Identification	Date	Initials	Injection Volume	Dilution	Comments
16	SG2673-2	4-23-13	KF	1mL	1	
17	↓ -3	↓	↓	↓	↓	
18	SG2471-5					RE-EXT
19	↓ -6					↓
20	Rinse					
21	Rinse	↓	↓	↓	↓	
1	Rinse	4-24-13	JH	1mL	1	
2	WG123104-1	↓	↓	↓	↓	
3	↓ -2					
4	↓ -3					
5	SG2571-4					
6	↓ -3					
7	↓ -5					
8	SG2707-1					
9	SG2571-6					
10	SG2707-2					
11	SG2711-1					
12	Rinse					
13	↓					
14	↓	↓	↓	↓	↓	
1	Rinse	4-25-13	JH	1mL	1	
2	WG123188-1	↓	↓	↓	↓	
3	↓ -2					
4	↓ -3					
5	SG2752-2	↓	↓	↓	↓	

Reviewed by: Review Date:

EX-011 – Revision 2 – 03/16/2010

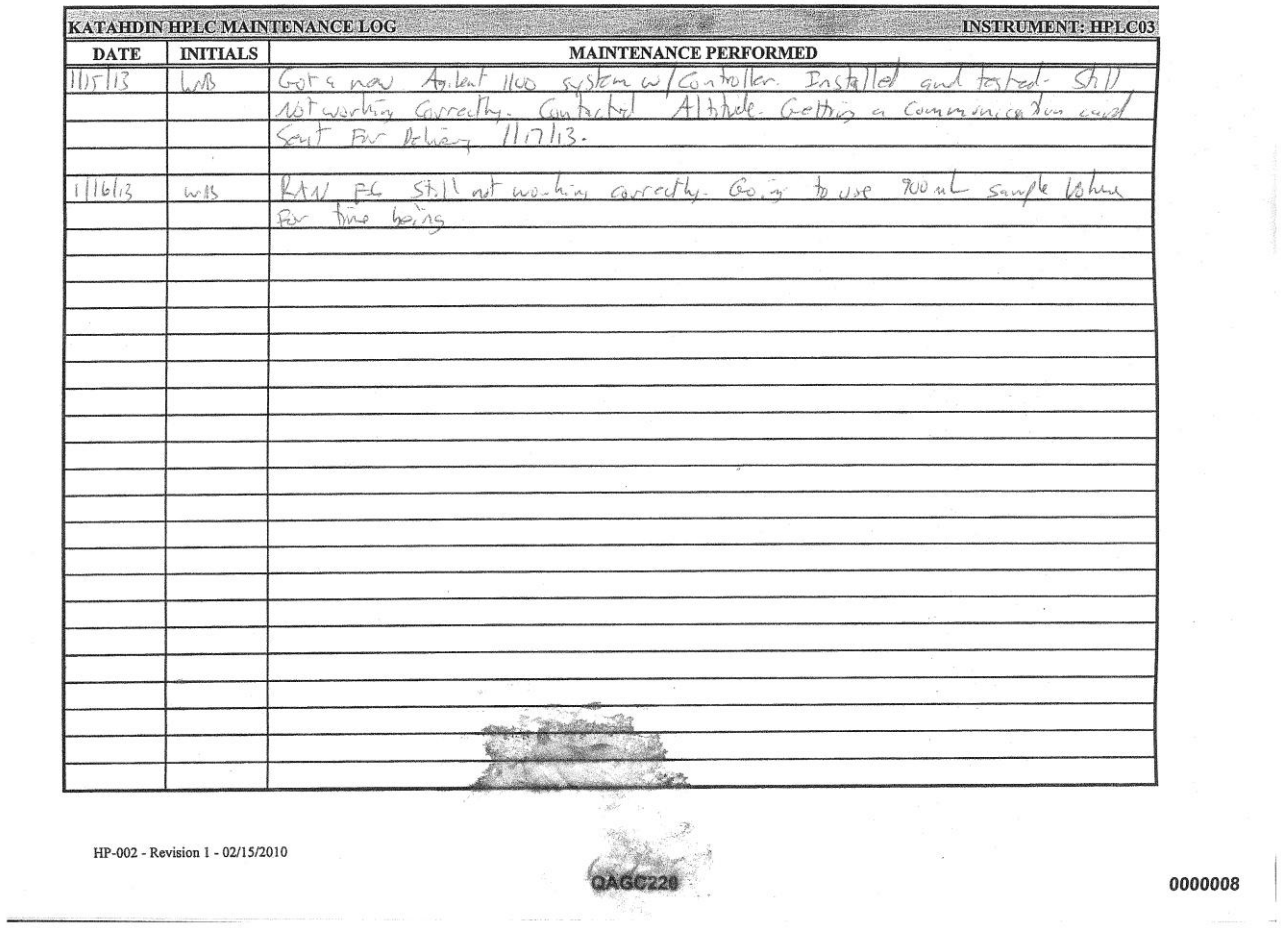
QAEX224

0000075

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP – EPH METHODS

FIGURE 4

EXAMPLE OF HPLC03 MAINTENANCE LOGBOOK PAGE



KATAHDIN HPLC MAINTENANCE LOG — INSTRUMENT: HPLC03

DATE	INITIALS	MAINTENANCE PERFORMED
11/5/13	WMS	Got a new Agilent 1100 system w/Controller. Installed and tested. Still not working correctly. Contacted Alltech. Getting a communication card and sent for delivery 11/7/13.
1/16/13	WMS	RAM PC still not working correctly. Going to use 900 uL sample volume for time being

HP-002 - Revision 1 - 02/15/2010

QAGC220

0000008

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 39°C to < 30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-108 reference for additional subsampling information.	LAD	02/09	02/09
08	Removed targeting sample weights. Added KAT SOP SD-902 reference. Updated logbook page example. Added GPC cleanup is required for all samples. Removed decanting samples	LAD	08/10	08/10
09	Minor modifications made to sections 5 & 7 to reflect current practices. Updated Section 9, to include LOD/LOQ requirements. Changed 7.6 & 7.7 to add surrogate and spikes after sodium sulfate is added. Updated references in Section 10.	LAD	04/12	04/12
10	Sect. 5 - Updated surr. prep. for SIM and Scan surr. - now in 1 mix. Sect. 7 - updated spiking info. for SIM/Scan surr. mix. Clarified decanting samples. Sect. 10 - Added & updated references. Updated fig. 1.	LAD	06/14	06/14
11	Sect. 4 and 7 - Updated for new sonicator. Changed KAS INC to KAS throughout.	LAD	08/15	08/15
12	Title changes for sections 1.4 + 5.0. Updated method references for NELAC, DOD + SW846. Removed old sonicater parameters. Removed a duplicate paragraph. Clarified sample weight.	LAD	09/17	09/17
13	Sect. 7 - updated method Blank and LCS initial weight criteria. Sect. 10 - updated references. Updated Logbook example	LAD	11/18	11/18

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Added use of tongue depressors. Corrected typographical errors.	CAD	04/19	04/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-14**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-14**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550C in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the

current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

An 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Pasteur pipets - disposable, 5 ¾ ".

4.3 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.4 Sonicator ultrasonic processor XL – QSonica Model Q500 (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.5 Vacuum filtration flask - 500 mL Erlenmeyer

4.6 Filter paper, 70 mm, Whatman #4

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Spatula - stainless steel

4.10 Wooden Tongue Depressors

4.11 Balance - capable of accurately weighing ± 0.01 g.

4.12 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent).

4.13 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.14 Powder funnels, 100 mm diameter, 35 mm stem

4.15 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.16 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.17 Nitrogen evaporation apparatus.

4.18 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

4.19 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS^{TM} with internal UV detection

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 200 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 SCAN/SIM Surrogate Spiking Solution - A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution -

Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.7 Base/Neutral and Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2.0 ug/mL for base/neutral and 4.0 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs

- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Sonicator horns tuned
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples should be cleaned using gel permeation chromatography (GPC) to reduce matrix interferences.

The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP SD-902, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples. Fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Do not decant any water on the sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2 Mix with a stainless steel spatula or wooden tongue depressor to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique “, for more detailed guidance on subsampling to ensure reproducibility.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out an approximate, greater than 30 g portion of sample into a

labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or wooden tongue depressor. Keep the spatula or wooden tongue depressor in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 $\pm$ 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **after** the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution. **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **after** the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 Turn sonicator on

7.9.1 Set pulse timer to 1 ½ minutes.

7.9.2 Set pulse to 1 and 1. This sets the sonicator to pulse for 1 second and rest for 1 second, with the timer set for 1 ½ minutes this equals a 3 minute total sonication

7.9.3 Set amplitude to 40%. Record this in the logbook.

7.9.4 These settings are stored in the unit and do not have to be entered with each use.

7.9.5 When done sonicating turn off the unit.

7.9.6 Refer to the Operating Manual for further information.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add approximately 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula or wooden tongue depressor to loosen up the mixture prior to extracting.

Rinse the spatula or wooden tongue depressor with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written in the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using approximately 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula or wooden tongue depressor. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 39°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.23 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.24 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.25 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.
Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3550C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The 2009 TNI Standards

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

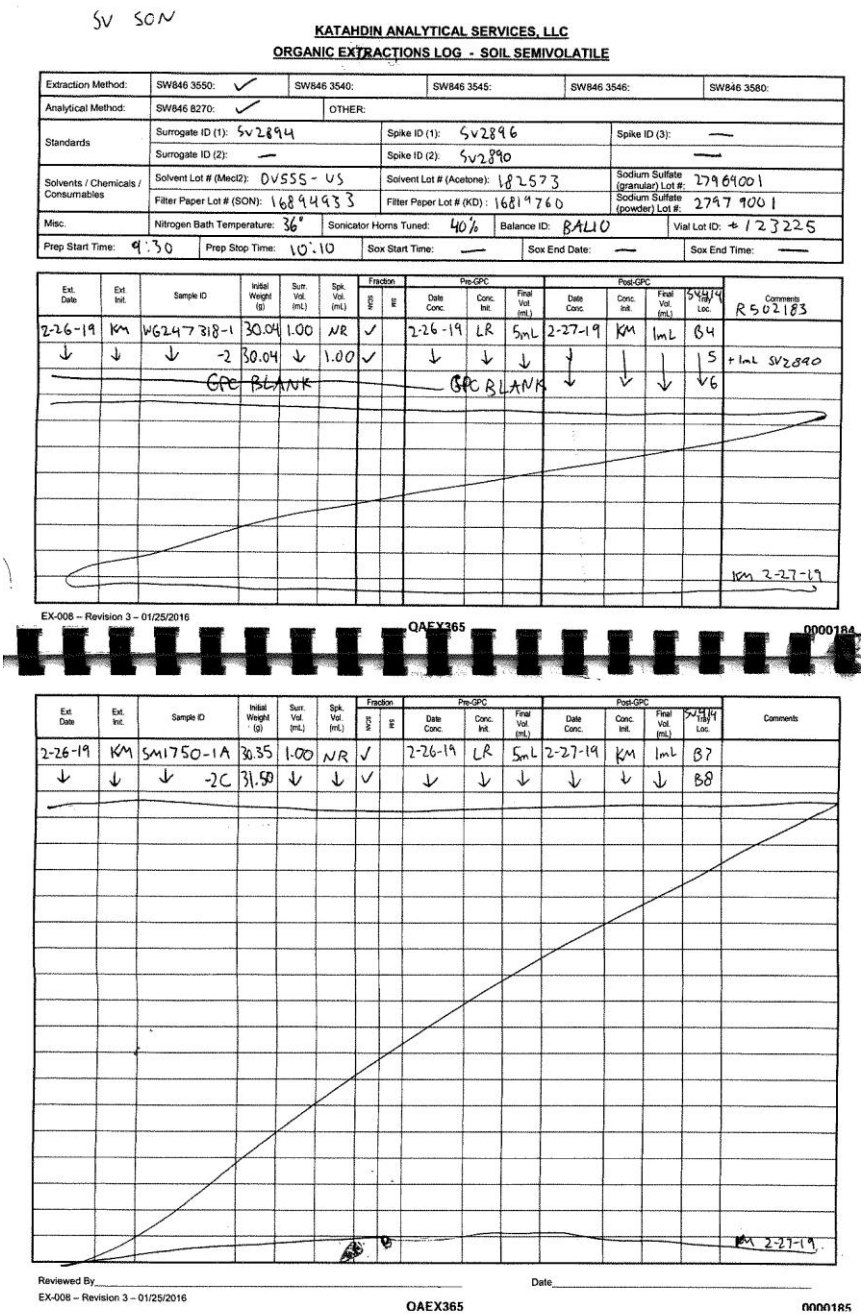
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-13	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV SON

KATAHDIN ANALYTICAL SERVICES, LLC

ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV2894		Spike ID (1): SV2896		Spike ID (3): —
	Surrogate ID (2): —		Spike ID (2): SV2890		—
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): DV555-US		Solvent Lot # (Acetone): 182573		Sodium Sulfate (granular) Lot #: 27989001
	Filter Paper Lot # (SON): 16894933		Filter Peper Lot # (KD): 16819760		Sodium Sulfate (powder) Lot #: 2797 9001
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: 40%	Balance ID: BAL10		Vial Lot ID: # 123225
Prep Start Time: 9:30	Prep Stop Time: 10:10	Sox Start Time: —	Sox End Date: —		Sox End Time: —

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WDS	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SVHH Trib/1 Loc.	Comments
2-26-19	KM	WG247318-1	30.04	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B4	R502183
↓	↓	↓ -2	30.04	↓	1.00	✓		↓	↓	↓	↓	↓	↓	5 V6	+ 1mL SV2890
		GPC BLANK							GPC BLANK						
															KM 2-27-19

EX-008 – Revision 3 – 01/25/2016 OAEX365 0000184

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WDS	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SVHH Trib/1 Loc.	Comments
2-26-19	KM	SM1750-1A	30.35	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B7	
↓	↓	↓ -2C	31.50	↓	↓	✓		↓	↓	↓	↓	↓	↓	B8	
															KM 2-27-19

Reviewed By_____ Date_____

EX-008 – Revision 3 – 01/25/2016 OAEX365 0000185

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JC Burton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.23.00

General Manager: Dennis P. Hogan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Miner changes throughout. clarifications to procedure section.	DN	10.23.00	
02	Addition of SPE Procedure Minor changes throughout Added wording to Sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques.	LAD	10/09	10/09
07	Added additional solvent exchange procedure. Updated logbook example.	LAD	08/10	08/10
08	Added that a MS/MSD should be extracted if enough sample volume and to extract an LCSD if NO MS/D. Added wording to H_2SO_4 prep. Minor changes to reflect current practices and remove duplication. Updated MDL-Sect. 9. Added and updated references. Removed method 3535 throughout. Changed PCB H.T to 30 Days w/ explanation.	LAD	04/12	04/12
09	Sect. 7- Removed the procedure of marking the sample meniscus on the sample bottle with a grease pencil.	LAD	05/13	05/13
10	Sect. 7- Nitrogen waterbath is set at 80°C, Several boiling stones are added to the conc. tube, CLLE are extracted for 18->24 hours, other minor edits for clarity. KAS name change.	LAD	12/14	12/14
11	Sect. 7- Removed requirement to check the pH of the sample prior to extracting.	LAD	08/15	08/15
12	Sect. 7- Added checking the sample pH prior to extracting to ensure it is between 5-9, and to adjust with acid or base if necessary.	LAD	04/17	04/17
13	Updated NAME of Sections 1.4 + 5.0. Updated method references for NELAC, DoD + SW846. Added residual chlorine testing. Removed MDL's performed every year from Section 2. Other minor revisions for clarity.	LAD	09/17	09/17

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 7 0 – Minor edits throughout to reflect current practices. Updated logbook example.	CAO	01/19	01/19

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-515-14,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-14,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel and continuous liquid-liquid extraction methods (EPA Methods 3510 and 3520).

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the “N-Hi” or “N-low” satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples

should be disposed of in the "O" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel or continuous liquid-liquid apparatus, following EPA Methods 3510 and 3520. The methylene chloride is exchanged with hexane for the final extract. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Stored in a Teflon capped glass bottle.

5.4 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4'-DDT	0.5
4,4'-DDD	0.5
4,4'-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.11 Potassium Iodide starch paper

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

The holding time for the extraction of aqueous pesticide samples for Methods 3510 and 3520 is 7 days from date of sample collection.

The holding time for the extraction of aqueous PCB samples by methods 3510 and 3520 is 30 Days

Note: SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit. The method recommends a holding time of 40 days from extraction to analysis for extracts stored under refrigeration in the dark; but also refers to SW846 Chapter 4, which specifies that there is no holding time for PCBs. Additionally, SW-846 states that the holding times listed in the method under the conditions listed (apparently referring to storage of extracts) may be as long as a year.

Holding times may be dictated by a project specific Quality Assurance Project Plan (QAPP), in a program specific Quality Systems Manual (QSM) or by a regulating body. If a project requires a holding time that is not specified above it must noted in the analysis notes of the workorder.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst

- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Prerinse all glassware as well as Teflon separatory funnels, caps and stopcocks three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook. Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.4 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples. An MS/MSD will be analyzed only

if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed. Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.7 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.8 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

7.9 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

7.10 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride to a clean sample container before adding to the separatory funnels for the blank and LCS/LCSD(s).

7.11 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.11 - 7.13). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.14 Repeat the extraction for a third time as described in 7.11 - 7.13.

7.15 Proceed to Section 7.28 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.16 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.17 Add several boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.18 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.19 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook. Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.20 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

7.21 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed.

7.22 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.23 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.24 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.25 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.26 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.27 Proceed to Section 7.28 for sample extract concentration procedures.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.28 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

7.29 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with approximately 15 mLs of methylene chloride and allow to drain. Add approximately 15 mL of hexane and allow to drain.

7.30 For methylene chloride extracts, add approximately 20 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.31 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).

7.32 Place the K-D in a hot water bath set at 75°C. Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane (methylene chloride for samples that have not gone through solvent exchange (ie. South Carolina samples). Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.33 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration.

7.34 Record the temperature of the nitrogen waterbath in the in the extraction logbook, also note any problems associated with the extract concentration.

7.35 Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.36 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap. Adjust final extract to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.37 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

7.38 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.39 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. An additional label is made for samples and method blanks to indicate which vial is the pesticide portion and which vial is the acid washed PCB portion. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of the analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 3510C and 3520C.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-14	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process (this modification is not allowed for samples originating from South Carolina).	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 2

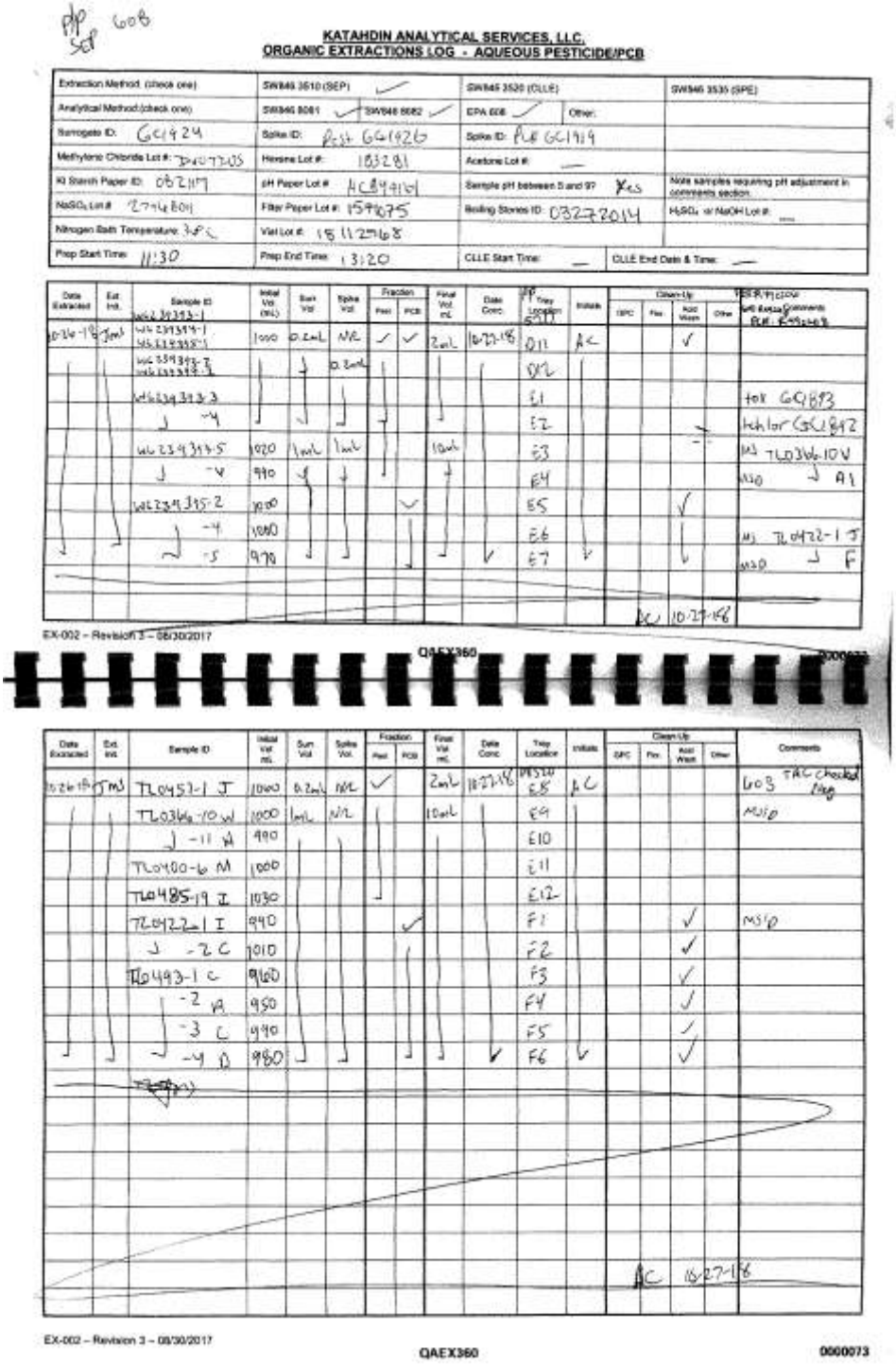
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-14	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process (this modification is not allowed for samples originating from South Carolina).	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES, LLC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3510 (SEP) ✓	SW846 3520 (CLLE)	SW846 3535 (SPE)
Analytical Method:(check one)	SW846 8081 ✓ SW846 8082 ✓	EPA 608 ✓ Other:	
Surrogate ID: GC1924	Spike ID: PCB GC1926	Spike ID: PCB GC1919	
Methylene Chloride Lot #: DW07205	Hexane Lot #: 183281	Acetone Lot #: —	
KI Starch Paper ID: 0622117	pH Paper Lot #: HC894161	Sample pH between 5 and 9? Yes	Note samples requiring pH adjustment in comments section.
$NaSO_4$ Lot #: 2794801	Filter Paper Lot #: 159675	Boiling Stones ID: 03272014	H_2SO_4 or NaOH Lot #: —
Nitrogen Bath Temperature: 34°C	Vial Lot #: 151127968		
Prep Start Time: 11:30	Prep End Time: 13:20	CLLE Start Time: —	CLLE End Date & Time: —

Date Extracted	Ext Init.	Sample ID	Initial Vol (mL)	Surr Vol	Spike Vol	Fraction: Pest	Fraction: PCB	Final Vol mL	Date Conc	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flo	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10-26-18	JM	WG339393-1 / WG339393-1	1000	0.2mL	NR	✓	✓	2mL	10-27-18	G11	AC			✓		
		WG339393-2 / WG339393-2			0.2mL					G12						
		WG339393-3								E1						for GC1893
		J -4								E2						Ichlor GC1892
		WG339395-1	1020	1mL	1mL			10mL		E3						MS TL0366-10 W
		J -4	990							E4						MSD J A1
		WG339395-2	1000				✓			E5				✓		
		J -4	1080							E6						MS TL0422-1 J
		J -5	970							E7	✓					MSD J F
												AC	10-27-18			

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Date Extracted	Ext. Init.	Sample ID	Initial Vol mL	Surr Vol	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol mL	Date Conc	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flo	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10-26-18	JM	TL0453-1 J	1000	0.2mL	NR	✓		2mL	10-27-18	PESTD E8	AC					608 TAC checked log
		TL0366-10 W	1000	1mL	NR			10mL		E9						MSD
		J -11 M	990							E10						
		TL0400-6 M	1000							E11						
		TL0485-19 I	1030							E12						
		TL0422-1 I	990				✓			F1				✓		MSD
		J -2 C	1010							F2				✓		
		TL0493-1 C	960							F3				✓		
		-2 A	950							F4				✓		
		-3 C	990							F5				✓		
		-4 D	980							F6	✓			✓		
												AC	10-27-18			

EX-002 – Revision 3 – 08/30/2017 QAEX360 0000073

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

Prepared By: Keith Tanguay Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 1/26/01

Operations Manager: Jol C. Burton Date: 1/26/01

QA Officer: Deborah J. Nadeau Date: 1.26.01

General Manager: Duncan F. Murfah Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8151A	Format changes, added pollution prevention, other minor changes to sections 7+8.	DM	1.26.01	1/26/01
02 8151A	Wording was added or changed to clarify sections 6, 7, 8 & 9. Minor changes throughout.	HRC	11.08.04	11.08.04
03 8151A	Sect. 7.0 - added information regarding TCLP samples. Small changes to reflect current practices. Updated Logbook page.	LAD	04/06	04/06
04 8151A	Sect. 7.4 & 7.10: changed glassware rinse solvent from ether to MeCl2. Sect. 7.5: Sample volume determined by comparison to reference bottle & added 800ml of DI water is added to TCLP samples. Sect. 7.10 & 7.11: time shaken from 4 to 3 min. 7.17: removed old way of determining sample vol. 7.18: Added it may be necessary to add more $MgSO_4$. 7.22: changed Vol, Sample Kd's to. 7.31: Added additional info to be recorded in logbook. Typos and formatting fixed.	LAD	03/08	03/08
05	Sect. 5.2 - Added wording to clarify acidification procedure. Sect. 7.13 - Removed necessity to reduce acid amt. for TCLP samples. Sect. 7.18 - added fume hood. Sect. 7.32 added $MgSO_4$ and NaCl2 lot #'s. Updated fig. 1 - logbook page.	LAD	05/09	05/09

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SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 7 and 10 - Added references. Sect. 7 - Added logbook requirements. Sect. 7.11 Changed manual shaking to mechanical shaker. Table 1 – Added method modification for storing acidified ether. Typographical errors corrected.	LAD	09/10	09/10
07	Minor changes made to section 7 to reflect current practices. Updated Section 9 and modified references in section 10. Added wording to H_2SO_4 preparation.	LAD	04/12	04/12
08	Sect. 5 - Changed standard prep solvent from acetone to methanol. Removed all other acetone references.	LAD	05/13	05/13
09	Sect-3- Removed rinsing the glassware and glasswool with acid. Changed KAS INC to KAS throughout.	LAD	08/15	08/15
10	Change title of section 5.0, Update method references for NELAC + DoD. Minor changes to sections 5.2, 5.3, 7.18 + 7.16.	LAD	09/17	09/17
11	Sect. 5- Removed checking pH of acidified $MeSO_4$ and how it is stored. Sect. 7- Changed phase separation to 3 minutes. Updated Logbook example	LAD	04/18	04/18
12	Sect. 7- minor edits to reflect current practices. Sect. 10 - Updated references. Updated logbook example.	LAD	03/19	03/19

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures, based on EPA SW 846 method 8151, used by Katahdin Analytical Services technical personnel for the extraction of chlorinated phenoxy acid herbicides from aqueous samples such as surface, well and discharge waters. Detection limits are at the ug/L level or greater

1.1 Definitions

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

DCAA: Dichlorophenylacetic acid

2,4-D: 2,4-Dichlorophenoxy acetic acid

ETHER: Diethyl ether- unpreserved

2,4,5-TP (Silvex): 2,4,5-Trichlorophenoxypropionic acid

2,4,5-T : 2,4,5-Trichlorophenoxyacetic acid

DIAZALD (a.k.a. Diazogen®): 99% (N-methyl-N-nitroso-p-toluenesulfonamide) See cautions in 1.3 Safety

CARBITOL: 2-(2-Ethoxyethoxy)ethanol

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of herbicides from aqueous samples. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of herbicides from aqueous samples to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

This procedure requires the use of materials that, if handled improperly, pose a potential health risk to everyone in the laboratory. Follow instructions that describe the use of commercially available peroxide test strips for Diethylether. Special care must be taken when working with diazomethane.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure. (See cautions prior to 7.24.)

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area

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2.0 SUMMARY OF METHOD

Chlorinated phenoxy acids and their esters are initially exposed to an alkaline hydrolysis at pH>12 and serially extracted three times with methylene chloride to remove chlorinated hydrocarbons and phthalate esters. The hydrolyzed sample then undergoes a pH adjustment to pH<2 and is extracted with diethyl ether. The diethyl ether extract is collected in a 500 mL screw cap bottle that contains approximately 20 grams of acidified anhydrous sodium sulfate. After drying for a minimum of two hours, preferably over-night, the extract is concentrated to 1 ml. The 1 ml extract is brought up to 4 mls with the addition of 1 ml of isooctane, 0.5 ml of methanol and 1.5 mls of diethyl ether. The 4 ml extract then undergoes diazomethane esterification (methylation) and is subsequently analyzed by GC-ECD. Compounds of interest are detected as methyl esters.

3.0 INTERFERENCES

Organic acids, especially chlorinated acids, cause the most direct interference. Phenols, including chlorophenols, also may interfere. Alkaline hydrolysis and subsequent extraction eliminate many of the predominant chlorinated insecticides. Because the herbicides react readily with alkaline substances, loss may occur if there is alkaline contact at any time except in the controlled alkaline hydrolysis step. Sodium sulfate (Na_2SO_4) should be acidified to minimize any alkaline contact.

4.0 APPARATUS AND MATERIALS

4.1 2 L Separatory Funnel, Teflon FEP with screw closures

4.2 Glass rod for crushing Na_2SO_4

4.3 pH paper (0-14)

4.4 gas tight volumetric syringes, 1.0 mL, 0.5 mL

4.5 mechanical separatory funnel shaker

4.6 Water/Steam bath (for K-D solvent evaporation) Organomation S-Evap Model 120

4.7 Kuderna-Danish apparatus:

- Concentrator tube (or collector), 10 mL graduated
- Evaporator flask, 500 mL
- Three ball macro Snyder column

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4.8 Nitrogen blow-down apparatus (for concentrating extracts in 10 mL concentrator tubes)

4.9 Pasteur pipets, Pasteur pipettes, 5 ¾"

4.10 12 mL vials

4.11 500 mL sample bottles

4.12 Scoopula(s)

4.13 Graduated cylinders, 25 mL, 100 mL, 1000 mL

4.14 Diazomethane Generator (See figure 2)

4.15 Boiling chips, teflon, or silicon carbide, (carborundum, 2 mesh)

4.16 Clean sodium sulfate jars (~2Liters) for collecting the aqueous phase.

5.0 REAGENTS AND STANDARDS

5.1 Potassium hydroxide (37%): Prepare by dissolving 37 g of potassium hydroxide pellets in DI water and diluting to 100 mL.

5.2 Acidified Anhydrous Sodium Sulfate: Prepare by adding hexane to a 2.5 Kg jar of sodium sulfate crystals until the crystals are completely submerged. Measure 25mL of concentrated hydrochloric acid or sulfuric acid with a graduated cylinder and add it to the hexane saturated salt crystals. Quickly stir the mixture with a glass rod until the sodium sulfate is loose. Then decant the solvent layer and transfer the sodium sulfate on to a sheet of aluminum foil under a hood. Let dry a minimum of 2 hours and then transfer back to the original jar. Label jar as acidified sodium sulfate. Record date and initials and the lot number for the acid used on the jar. Cover and store at room temperature. Acidified anhydrous sodium sulfate will be referred to as sodium sulfate further in this SOP.

5.3 Sulfuric acid (1:3 H_2SO_4 : H_2O): Prepared in an icebath by slowly adding 250 mL of concentrated H_2SO_4 to 750 mL of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic. Store the diluted acid in a laboratory refrigerator.

5.4 Herbicide surrogate solution containing 5 mg/mL DCAA acid in methanol.

5.5 Underivitized Chlorinated Herbicide Stock Solutions: Contains 18 compounds at various concentrations - 100 ug/ml for all except for MCPA and MCPP which are at 10,000 ug/ml. Dilute to 5.0 ug/ml and 500 ug/ml with methanol.

5.6 Diazald solution: weigh out ~6.8 grams of Diazald into a 50 mL volumetric flask and dissolve in a mixture of 25 mL carbitol and 25 mL of ether (50 mL of 1:1 v/v carbitol/ether). Bring to the volume mark only after all of the Diazald is in solution. Use the sonicator bath briefly if necessary. This solution is stable if kept at -10 - 20^{o}C for one month.

5.7 Ether: pesticide residue grade or equivalent (unpreserved).

5.8 Methanol: pesticide residue grade or equivalent.

5.9 Carbitol: 2-(2-Ethoxyethoxy) ethanol

5.10 Hexane: pesticide residue grade or equivalent

5.11 Isooctane: pesticide residue grade or equivalent

5.12 Organic-free reagent water.

5.13 Silicic Acid: (H_2SIO_5) - 100 mesh powder.

5.14 10N Sodium Hydroxide

5.15 Sodium Chloride: (NaCl) - Pre-baked at 400°C for at least 4 hours.

5.16 Laboratory Reagent Grade Water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are collected in a 1L amber glass bottle. Samples are stored at 4 (±) °C until extraction.

Holding time for extraction of aqueous samples for Method 8151 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

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7.0 PROCEDURES

INITIAL EXTRACTION

Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Extraction and concentration dates
- Extraction and concentration analyst
- Separatory funnel extraction start and end times.
- Sample ID or QC sample ID
- Initial and final volumes
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

7.1 Assemble, label, and methylene chloride rinse a 2 liter separatory funnel with stopcock and closure, a 500 mL glass sample bottle, and a 2 Liter Na_2SO_4 jar for each sample including blank, lab control sample and lab control sample duplicate. Make sure no residual $MeCl_2$ is present on glassware.

7.2 Assign a separate quality control number to each blank and associated spike and record this information in the appropriate log book. One blank and at least one LCS should be extracted per batch.

7.3 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirement. When the client does not specify sample QC, then the extractions lab will chose a sample for quality control (one set per 20 samples designated as MS/MSD) to extract and analyze. If sufficient volume of sample(s) is not available for an MS, MSD, the lab will extract an LCS, LCSD instead.

7.4 Determine sample bottle volumes by comparing to reference bottle. Record sample volumes in logbook. Transfer the 1-L sample aliquot to a 2-L separatory funnel. For TCLP samples, transfer 200ml of sample to funnel using a 6.5oz plastic

graduated cup. 800mL laboratory reagent grade water is added to all TCLP samples.

7.5 Laboratory reagent grade water will serve as the method blank and lab control sample (LCS). For each blank and LCS, add 1000 mL of DI water to the separatory funnel using a clean 1000 mL graduated cylinder. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 Using a gas-tight volumetric syringe, add 1mL of herbicide surrogate solution to all of the samples, including blank and LCS's. Record surrogate number and amount added. Rinse syringes with methanol before and after use. Extreme accuracy should be used when measuring and adding spike and surrogate solutions! Double check the solution number and amount used.

7.7 Add 1mL herbicide spiking solution to LCS and MS/MSD, as required. Record spike number and amount added in appropriate logbook. Rinse syringes with methanol before and after use.

7.8 Add 250g of NaCl to the samples and the QC. Seal and shake to dissolve the salt.

Hydrolysis

7.9 Add 17 mL of 10N NaOH to all 1-L samples (TCLP and regular). Using pH paper, adjust the pH to 12 or greater by adding more 10N NaOH if necessary. Shake for 3 minutes on mechanical shaker. Let samples sit for ~ 15 minutes, shake for 3 minutes and let sit for ~ 15 minutes. Repeat for shake/sit for at least 1 hour. This will complete the hydrolysis step.

Solvent Washes

7.10 Add ~60mL of methylene chloride to the sep funnel. Extract the sample by vigorously shaking for 3 minutes on mechanical shaker. Allow the organic layer and the water layer to settle until there is a clear separation of the 2 phases. Discard the methylene chloride layer.

7.11 Repeat step 7.11 two more times, discarding the methylene chloride layer each time.

Extraction

7.12 Add 17 mL of cold (4°C) 1:3 sulfuric acid to all samples, seal, and shake to mix. Using pH paper, adjust the pH of the sample to ≤2 by adding more 1:3 sulfuric acid

if necessary. After adding acid, discard any residual methylene chloride which may settle out.

7.13 Add 120 mL ether to each separatory funnel. Shake and vent until there is no more pressure build-up. Shake on mechanical shaker for 3 minutes. Allow the phases to to settle until there is a clear separation of the 2 phases.

7.14 Collect the aqueous (bottom) layer in a 2 liter Na_2SO_4 jar, and the ether (top) layer in a 500 mL sample bottle containing about 20g of acidified Na_2SO_4. Cap and shake the ether layer and drying agent. Return the aqueous layer to the separatory funnel.

7.15 Extract the aqueous layer two more times with 60 mL aliquots of ether as in steps 7.14-7.15. Combine ether (top layer) in the sample bottle. Prior to last extraction, rinse the 2-L Na2SO4 jar with ether and transfer to funnel to remove any remaining analytes.

7.16 Dispose the aqueous layer in the “N low” waste (sep-funnel waste) container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood overnight or rinse them with tap water before bringing to dish washing area.

Drying Step

7.17 Additional acidified Na_2SO_4 is added to the extract if it is not free flowing crystals or it is in a cake form. Shake the extract (ether phase) and the drying agent for one minute.

7.18 Allow the extract to remain in contact with the Na_2SO_4 for at least two hours, but, preferably stored overnight in the fume hood.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. Dispose of waste in the N-Low Sep. Funnel waste container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area.

Extract Concentration

7.19 Assemble a Kuderna-Danish apparatus with concentrator tube for each sample. Rinse the KD glassware with methylene chloride followed by ethyl ether making sure no residual solvent is present on glassware before transferring samples.

7.20 Carefully, decant the extract and the acidified sulfate from the amber jar. Filter the sample though the acidified sodium sulfate crystals into the K-D apparatus. Avoid allowing any acidified sodium sulfate crystals to fall in the concentration tube. Use a glass rod to crush any caked sodium sulfate in the glass jar. Rinse the glass jar 3 times with 10 mL of ether. Let drain between rinses. Thoroughly rinse funnel with ether and let drain.

7.21 Add 2 clean boiling chips to the K-D collector and attach a Macro-snyder column. Pre-wet the column with ether and place the K-D apparatus on the steam bath (which is heated no higher than 60°C). When the volume of liquid reaches approximately 2-4 mL, remove from the steam bath and allow to drain and cool for several minutes. Use caution, the ether will evaporate rapidly!

7.22 Remove the column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of ethyl ether.

7.23 Concentrate the sample to 1.0 mL using the nitrogen blow-down apparatus.

7.24 Add 1.0 mL of isooctane and 0.5 mL of methanol. Rinse pipette and sides of tube to dilute to final volume of 4.0 mL with ether.

Diazomethane Esterification

VERY IMPORTANT: Diazomethane is a toxic carcinogen and can explode under certain conditions! The following precautions MUST be followed:

- Use only a well ventilated hood. Do not breathe the vapors.
- Do not heat above 90 ºC. Explosion may result.
- Diazomethane must NEVER come into contact with ground glass surfaces as rough surfaces are proven initiators of detonations.
- Avoid exposure of the solution to bright light - explosion may result.
- Always use EXTREME caution when handling either diazomethane or Diazald.

7.25 Assemble the diazomethane bubbler (see Figure 2).

7.26 Add 5.0 mL of ether to tube l. Add about 2.0-3.0 mLs of the Diazald solution (from 5.6) and 1.5 mL of 37% KOH to tube 2. Add the 37% KOH solution last to begin the reaction, and quickly cap both tubes.

7.27 Apply a nitrogen flow of 5-10 mL/min so bubbles emerge slowly. Bubble blank and LCS's before samples. Bubble directly into the sample extract's concentrator tube. Allow the diazomethane to bubble through the sample for 45-60 seconds or until the yellow color persists. **Note: There will be no yellow color for TCLP samples.**

There is sufficient Diazald solution for esterification of two, maybe three, samples. Rinse exit tube with ether between samples.

7.28 Remove the exit tube form the KD concentrator tube, cover and store at room temperature for 20-30 minutes.

7.29 Destroy any unreacted diazomethane by adding 0.1 to 0.2 g silicic acid to the collectors. Allow to stand until the evolution of nitrogen is complete. Pipette the extract into a hexane rinsed 12 mL vial and rinse the collector twice with hexane (1-2 mL each time). Be careful to leave the silicic acid in the concentrator tubes. Bring to a final volume of 10mL using a reference vial, mix well, and let sit for a few minutes.

7.30 The extract is now ready for analysis. Make sure that initial volumes, intermediate volumes, aliquot volumes, and final volumes have been recorded in the logbook. Also verify that the surrogate and spike identification numbers and amounts used have been recorded as well as the Diazald identification number. The temperature of the water in the nitrogen evaporation water bath is recorded in the extraction logbook. The lot numbers of all of the solvents, acids and bases, sodium sulfate, sodium chloride, as well as all of the filter papers that are used in the extraction process are recorded in the logbook. Any deviations from the SOP or any abnormal sample observation should be noted as comments.

7.31 The data entered here are later used in calculations of the final result; see SOP CA-305, Analysis of Herbicides in Extracts of Water & Soil.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 The purity of the solvents used is checked on a lot number basis and is kept on file in the Extractions Laboratory.

8.2 A method blank and a laboratory control sample (LCS) must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each sample matrix (water)
- Each extraction method or level
- Every batch of 20 samples, or fewer, extracted in a 24-hour period

8.3 A matrix spike (MS), and matrix spike duplicate (MSD) should be prepared every 20 samples.

8.4 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirements. When the client does not specify sample QC, the extractions lab will choose one (per 20) samples for quality control to extract and analyze.

8.5 Surrogate and Spike acceptability criteria can be found in SOP CA-305, Analysis Of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151.

8.6 If all quality control criteria are not met, appropriate steps must be taken to determine the cause. Problems indicate either matrix effect or an out of control event in the procedure.

8.7 Batch QC Requirements: If surrogates or spike compounds fail their criteria in the blank or lab control samples, the entire extraction batch is in question. A Corrective Action Report is initiated in the GC lab, and completed using information obtained from the Organics Prep Lab. If possible, the entire batch of samples is re-extracted with new QC samples. If no more sample can be obtained for re-extraction, any results reported must be flagged in the report and a narrative is included qualifying the data. Refer to SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, for further details.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO
Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

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10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8151A.

Katahdin SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation NELAC Derivatization: SW-846 Method 8151, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-516-12	Method 8151, current revision
Apparatus/Materials	Use nitrogen blowdown technique.	Use two ball micro Snyder column.
Reagents	Unpreserved diethyl ether. 10N NaOH. Acidify sulfate in Hexane and store at room temperature.	Ethyl ether preserved with BHT. 6N NaOH. Acidify sulfate in Ether and store at 130 ºC.
Sample preservation/ handling		
Procedures	Shake for 3 minutes. Samples poured through acidified sulfate before KD.	Shake for 2 minutes. Samples poured through acidified glass wool before KD.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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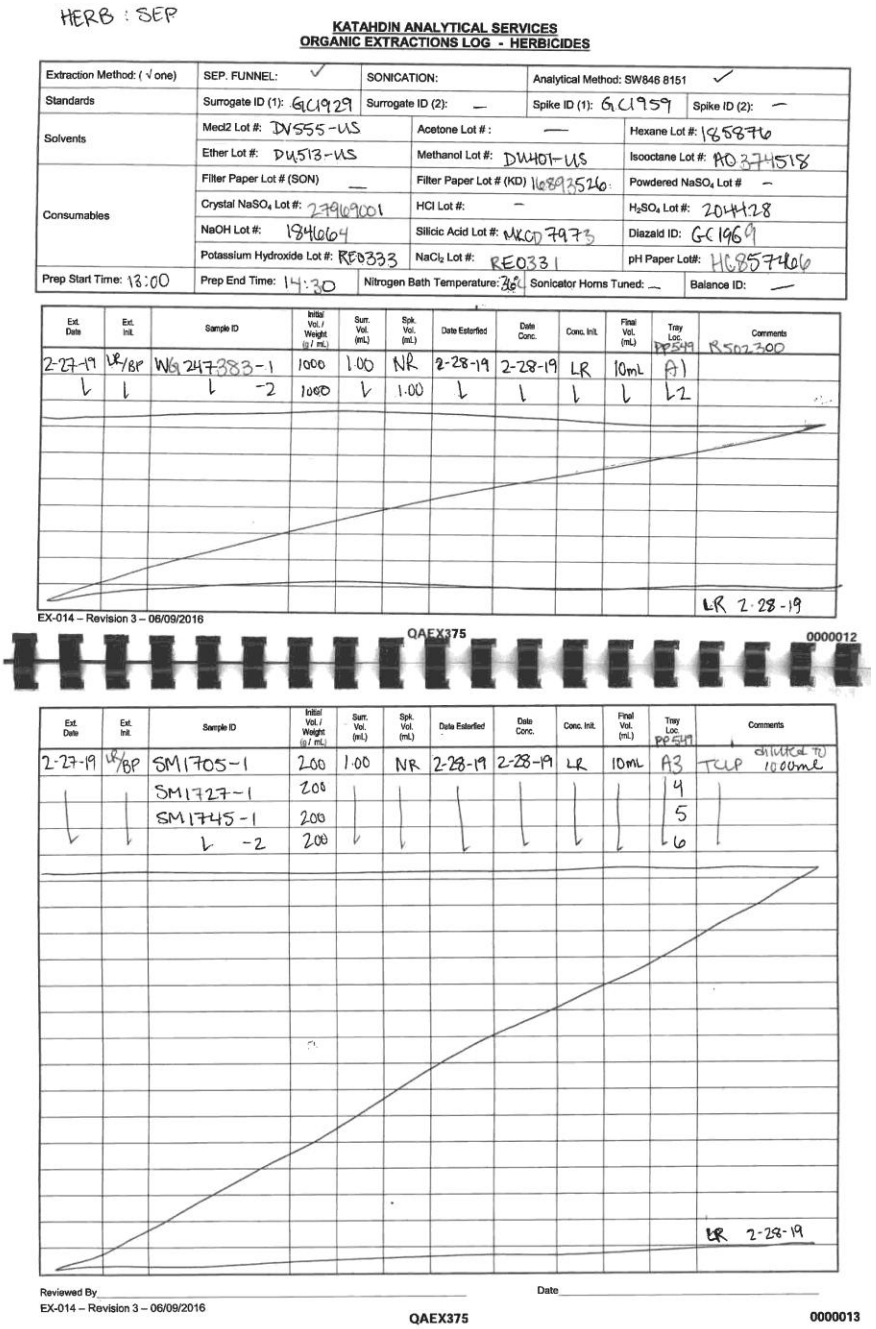
FIGURE 1

EXAMPLE OF AQUEOUS HERBICIDE LOGBOOK PAGE



HERB : SEP

KATAHDIN ANALYTICAL SERVICES
ORGANIC EXTRACTIONS LOG - HERBICIDES

Extraction Method: (√ one)	SEP. FUNNEL: ✓	SONICATION:		Analytical Method: SW846 8151 ✓	
Standards	Surrogate ID (1): GC1929	Surrogate ID (2): —		Spike ID (1): GC1959	Spike ID (2): —
Solvents	MeCl2 Lot #: DV555-US	Acetone Lot # : —		Hexane Lot #: 1858796	
	Ether Lot #: DU513-US	Methanol Lot #: DUH01-US		Isooctane Lot #: A0374518	
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) 168935260		Powdered $NaSO_4$ Lot # —	
	Crystal $NaSO_4$ Lot #: 23969001	HCl Lot #: —		H_2SO_4 Lot #: 2044128	
	NaOH Lot #: 184664	Silicic Acid Lot #: WKCD7973		Diazald ID: GC1969	
	Potassium Hydroxide Lot #: RE0333	$NaCl_2$ Lot #: RE0331		pH Paper Lot#: H6857406	
Prep Start Time: 13:00	Prep End Time: 14:30	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: —	Balance ID: —	

Ext. Date	Ext. Init.	Sample ID	Initial Vol./ Weight (g / mL)	Surr. Vol. (mL)	Spk. Vol. (mL)	Date Esterified	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc. PP549	Comments
2-27-19	LR/BP	WG247383-1	1000	1.00	NR	2-28-19	2-28-19	LR	10mL	A1	R502300
↓	↓	↓ -2	1000	↓	1.00	↓	↓	↓	↓	↓2	

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0000012

Ext. Date	Ext. Init.	Sample ID	Initial Vol./ Weight (g / mL)	Surr. Vol. (mL)	Spk. Vol. (mL)	Date Esterified	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc. PP549	Comments
2-27-19	LR/BP	SM1705-1	200	1.00	NR	2-28-19	2-28-19	LR	10mL	A3	TCLP diluted to 1000mL
↓	↓	SM1727-1	200	↓	↓	↓	↓	↓	↓	4	↓
↓	↓	SM1745-1	200	↓	↓	↓	↓	↓	↓	5	↓
↓	↓	↓ -2	200	↓	↓	↓	↓	↓	↓	6	↓

LR 2-28-19

Reviewed By_____ Date_____

EX-014 – Revision 3 – 06/09/2016

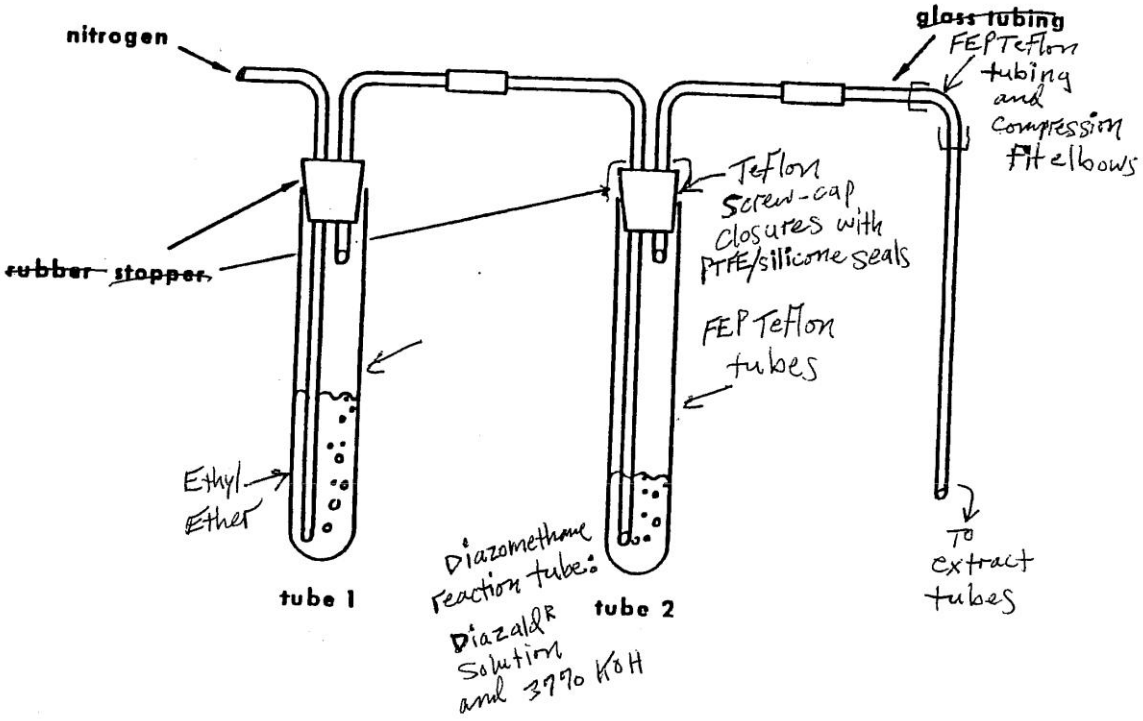
QAEX375

0000013

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

FIGURE 2

DIAZOMETHANE SOLUTION GENERATOR



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Melissa Rosa

Review Date: 1-04-20

SOP Number: CA-516-12

SOP Title: Preparation of Aqueous samples for herbicides by Method 8151

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-29-2020

QAO Signature: Lucie Dimmel **Date:** 020520

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.15.00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	JM	11.15.00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09
07	Removed targeting sample weights. Removed decanting samples prior to extraction.	LAD	08/10	08/10
08	Minor changes to section 7 to reflect current practices. Updated section 7.6 for frequency of MS/D's. Added information for MDL, LOD and LOQ to Section 9. Updated references. Added H_2SO_4 to reagents.	LAD	04/12	04/12
09	Sect. 7 - Water bath temp. is set at 80°C, fixed typo, added information @ decanting, Sect. 10 - Updated references. Updated KAS INC. to KAS LLC. Updated fig. 1.	LAD	12/14	12/14
10	Sect. 5 - Added additional LCS/MS spikes. Sect. 6 - Changed PCB H.T. to 30 days. Sect. 7 - Small edits for clarity and to reflect current practice. Added Attachment A - Total Solids Determination	LAD	01/18	01/18
11	Added wooden tongue depressors, Corrected rheostat setting. Updated references. Updated logbook page example	LAD	03/19	03/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

I acknowledge receipt of copy ___ of document **SOP CA-524-11**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste

stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

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4.1.1 500 mL flat-bottom boiling flask

4.1.2 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Gastight Volumetric Syringes – Various sizes

4.15 Top-loading balance - capable of weighing to 0.01 g.

4.16 Spatulas, stainless-steel

4.17 Wooden Tongue Depressors

4.18 Long forceps, stainless-steel

4.19 Metal clips – for securing Soxhlets to boiling flasks

4.20 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.21 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Sulfuric acid solution (1:1 H2SO4 : H2O) – Prepared in an icebath by slowly adding a volume of concentrated H2SO4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.4 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.5 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.7 Matrix Spike/Lab Control Sample spiking solution

5.7.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone

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Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.7.2 Aroclor 1660 spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7.3 Aroclor 1254 spike solution – prepare Aroclor 1254 in pesticide grade acetone at a concentration of 5.0 ug/mL.

5.7.4 Technical Chlordane spike solution and Toxaphene spike solution – prepare separately, in pesticide grade acetone at a concentration of 10 ug/mL each.

5.7.5 Additional miscellaneous pesticide compounds – Prepare in pesticide grade acetone at a concentration of 0.5 ug/mL each

5.8 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

The holding time for extraction of Pesticide sediment/soil samples by Method 3550 is 14 days from date of sample collection.

The holding time for PCB only sediment/soil is 30 days. SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit.

Samples extracted for Pesticide and PCB must be extracted within the 14 day holdtime.

Analysts should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

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7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Extraction start and end times
- Soxhlett extraction start time and end time and date
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Do not decant any water on the sediment sample

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Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2.2 Mix the sample thoroughly with the stainless steel spatula or wooden tongue depressor. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.3 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.4 Refer to Attachment A for total solids determination.

7.2.5 Please refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Techniques" for more information of subsampling.

7.3 Weigh out an approximate, greater than 30 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or wooden tongue depressor. Keep the spatula/tongue depressor in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB

analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set should be prepared for every 20 samples. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed. To prepare MS/MSD, weigh out two approximate, greater than 30g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas or wooden tongue depressors. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful that none of solid material falls into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 40-45% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 16-24 hours. Be

sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as much solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 Pest/PCB and Pesticide only extracts will be GPC'd unless there is a time constraint.

If extracts are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. **Extracts that will be GPC'd are not solvent exchanged into hexane.**

7.15 All extracts (GPC and non-GPC) - Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask

three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.16 Extracts not being GPC'd – Begin the solvent exchange of the extracts by adding approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.16.1 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.17 All extracts (GPC and non-GPC) – Please use the same solvent as the extract for rinsing and bringing the extract to the final volume. Methylene chloride should be used with extracts that are to be GPC'd. Hexane should be used for extracts the either have been GPC'd or are not going to be GPC'd.

7.17.1 Place the K-D in a hot water bath set at 75°C. Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane or methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane or methylene chloride.

7.17.2 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.17.3 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane or methylene chloride extract to 10 mL (or a client specified final volume) in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.17.4 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Record the box number and "tray location" for each extract vial.

7.18 Additional cleanup for non-GPC'd extracts:

7.18.1 Extracts for 8082 PCB only analysis must be sulfuric acid washed. The associated Method Blank and LCS must also be sulfuric acid washed. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA-525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

7.18.2 Extracts for combined 8081/8082 analyses must be split. Mix extract well before splitting. One portion, including the Blank, LCS and MS/MSD, must be sulfuric acid washed for 8082 analysis. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD’s must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.
MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-11	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB SOX

KATAHDIN ANALYTICAL SERVICES
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550	SW846 3540 ✓	SW846 3545	SW846 3546	SW846 3580
Analytical Method: (check one)	SW846 8081	SW846 8082 ✓		EPA 608	
Standards	Surrogate ID: GC1953	Spike ID: GC1946		Spike ID: —	
Solvents	Solvent Lot # (MeCl2): DV855-US	Solvent Lot # (Acetone): —		Solvent Lot # (Hexane): 185876	
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) 16819760	Acid Lot # 204428	Boiling Chip Date: 112618	
	Na_2SO_4 (granular) Lot #: 279.6900	Na_2SO_4 (powder) Lot #: 279 78003	Vial Lot # 18112968	—	
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Amplitude: —		Balance ID: BAL 10	
Prep Start Time: 12:45	Prep End Time: 13:15	Soxhlett Start Time: 13:55		Soxhlett End Date & Time: 7:55 1-24	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	PP543 Tray Loc.	RU99920 Comments
1-28-19	KM	WG245310-1	30.01	1.00	NR		✓				✓	1-29-18	KM	10mL	D6	
↓	↓	↓ -2	30.03	↓	1.00		✓				✓	↓	↓	↓	D7	

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	PP543 Tray Loc.	Comments
1-28-19	KM	SMO753-1A	31.88	1.00	NR		✓				✓	1-29-18	KM	10mL	D8	
		-2	31.94												D9	
		-3	31.17												D10	
		-4	30.86												D11	
		-5	30.46												D12	
↓	↓	-6 ↓	31.82	↓	↓		↓				↓	↓	↓	↓	E2	

KM 1-29-19

Reviewed By_____ Date_____

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

ATTACHMENT 1

DETERMINATION OF PERCENT DRY WEIGHT

When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed within 14 days of the sample date.

WARNING: The drying oven should be contained in a hood or be vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

1.0 PROCEDURES

1.1 In accordance with the current revision of Katahdin SOP CA-102, “Balance Calibration”, verify balance calibration at the beginning of the day or prior to beginning analysis. Refer to Section 8.0 for acceptance criteria.

1.2 The oven ID must be recorded in KIMS

1.3 Label and weigh each tin dish before use and record initial (tare) weight in KIMS.

1.4 Samples will be analyzed under this procedure as “total solids” or “decanted total solids”. “Total Solids” will be performed on a well-homogenized sample as received where any aqueous phase is incorporated into the solid matrix prior to aliquotting. “Decanted total Solids” will be performed on sample aliquots designated for extractable organic parameters where it has been determined that the standing aqueous layer must be decanted prior to aliquotting for the organic extractable preparation. Sample login decants the samples during the login process. Decanted samples will marked with a “DEC” on the cap. The decanted sample aliquot will constitute the “original” sample for total solids determination.

1.5 Whether the sample has been decanted or not, remove any obvious non-representative material such as large sticks and rocks. Thoroughly mix the sample with a scupula. Weigh 10-20 g of a representative sample aliquot into a labeled weighing dish. For samples that are analyzed “as received” and contain a significant standing aqueous layer, a larger aliquot may be used. In order to facilitate drying and minimize moisture entrainment, the sample may be spread out across the surface of the pan/dish/crucible. Please refer to Katahdin SOP, CA-108, Basic Laboratory Technique, current revision, for more information on subsampling.

1.6 Record the weight (to the nearest 0.0001g) of the sample plus dish by uploading the weight directly into KIMS. Please refer to the current version of SOP CA-717, Total Solids/Total Volatile Solids Determination in Solid Matrices, for instructions.

1.7 A Method Blank and LCS are prepared at a minimum frequency of 1/20 field samples and a Duplicate is prepared at a minimum frequency of 1/10 field samples or per preparation batch if fewer samples.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.8 A Method Blank consists of a weighing tin/dish/crucible spiked with 0.50 mL of water carried through the entire analytical process. The Method Blank result must be less than the PQL of 0.1 wt%. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

1.9 The LCS is prepared by spiking muffled dry sand with a known amount of laboratory reagent grade water. Weigh out 4.5 g of sand and 0.50 mL (0.50 g) of laboratory reagent grade water and carry LCS through entire analytical process. Recovery for the LCS must be within 90-110% of the expected true value. If acceptance criteria are not met, all associated samples must be reanalyzed if possible.

1.10 A Duplicate is prepared by taking a duplicate sample aliquot, 10-20 g, through the procedure as described for the sample. NOTE: The duplicate should be aliquotted at the same time as the original sample.

1.11 Transfer samples to drying oven maintained at 103-105°C. Record “date in”, “time in”, and “temperature in” in KIMS.

1.12 Dry samples for greater than 12 hours. Remove the samples from the oven and cool to ambient temperature in a desiccator. Record “date out”, “time out”, and “temp out” in KIMS.

1.13 Weigh and record in KIMS the final sample plus dish weights to the nearest 0.0001g.

1.14 The raw data (batch number, sample number, pan weight, original sample + pan, and final sample + pan weights) are entered into KIMS for calculation. The calculation performed is as follows:

$$\%\ \text{Total Solids} = \frac{(\text{B} - \text{pan, g})}{(\text{A} - \text{pan, g})} \times 100$$

Where: *A* = weight of original sample+ pan, g, and
B = weight of dried sample + pan, g

1.15 EXPORTING TOTAL SOLIDS DATA INTO KIMS – Please refer to the current version of SOP CA-717, Total Solids/Total Volatile Solids Determination in Solid Matrices, for instructions.

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: JR Brennan

Review Date: 1-20-20

SOP Number: CA-524-11

SOP Title: Soxhlet Extraction P/P 3540

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-20-2020

QAO Signature: Lucie Dimond **Date:** 01.21.20

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.16.00

General Manager: [signature] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 & 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LMD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~spikes~~ compounds used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added additional compounds to Fig. 2. Added recording of consumable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised Section 7 to reflect current techniques. Added SOP CA-108 reference for Sub-Sampling. Updated logbook example	LAD	08/09	08/09
07	Removed targeting sample weights. Added to weigh out a minimum 30g sample. Removed decanting samples prior to extracting. Added putting solid waste in the "I" waste Stream. Updated Logbook page.	LAD	08/10	08/10
08	Section 5.5 - Added 1.4-Dioxane-d8 and updated 2.4-Dibromophenol's concentration. Section 5.7 - Updated acid spikes concentration. Sections 7.0, 7.6 and 7.13 - minor changes to reflect current practice. Added MDL, LOD and LOQ information to Section 9. Updated references in Section 10.	LAD	04/12	04/12
09	Sect. 5 - Updated Surr. prep. for both SIM and Scan Surr. - now in 1 mix. Sect. 7. Updated spiking info. for SCAN/SIM surr. Clarified decanting soils. Sect. 10 - Added and updated references. Updated Fig. 1.	LAD	06/14	06/14
10	Sect. 1 - Added pollution control to Waste Disposal Sect 5 - Added standards to title. Sect. 7 - Added to record the date Sox. ends, changed M. BLK and LCS initial weigh from >30g to 30g. Updated Fig. 1 - logbook ex. KAS INC → KAS throughout	LAD	08/16	08/16
11	Updated method references for DOD+SW846. Clarified weight of sample. Fixed grammatical errors. Changed water bath temperature.	LAD	09/17	09/17
12	Added Wooden tongue depressors, updated Rheostat setting, updated references, Corrected typographical errors	LAD	04/19	04/19

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-526-12**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-526-12**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Documentation of Capability”.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream

satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

4.1.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.2 500 mL flat-bottom boiling flask

4.1.3 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Wooden Tongue Depressor

4.17 Long forceps, stainless-steel

4.18 Metal clips – for securing Soxhlets to boiling flasks

4.19 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, pre-baked, certified by the manufacturer/vendor.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 SV SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral and 4 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Extraction and concentration dates
- Extraction and concentration analyst
- Soxhlet extraction start and end dates and times
- Prep Date and start and end times.
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

Pre-rinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Do not decant any water layer on a sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2.2 Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, "Basic Laboratory Technique" for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out an approximate, greater than 30 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30 g ±0.5g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ±0.5 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If

combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SV SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on

the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 40-45% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as much solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures along with the glass wool from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries. Put the solid waste in the "I" waste stream.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative

transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality

Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

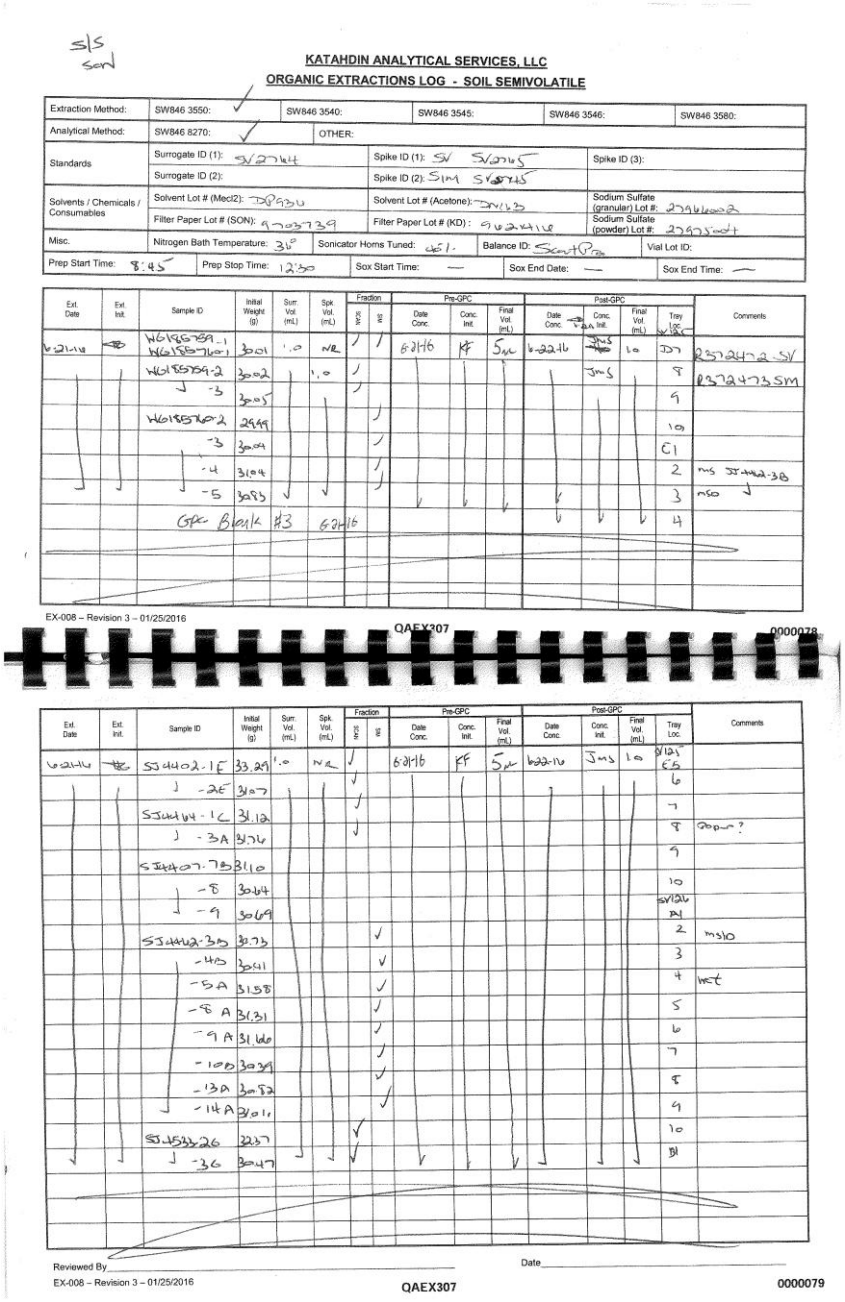
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-12	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES, LLC

ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV 27164	Spike ID (1): SV SV 27165	Spike ID (3):		
	Surrogate ID (2):	Spike ID (2): SIM SV 27145			
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): DP9314	Solvent Lot # (Acetone): DN163	Sodium Sulfate (granular) Lot #: 279616 w2		
	Filter Paper Lot # (SON): 97037139	Filter Paper Lot # (KD): 96244168	Sodium Sulfate (powder) Lot #: 279515 w1		
Misc.	Nitrogen Bath Temperature: 35°	Sonicator Horns Tuned: ✓	Balance ID: Scout Pro	Vial Lot ID:	
Prep Start Time: 8:45	Prep Stop Time: 12:30	Sox Start Time: —	Sox End Date: —	Sox End Time: —	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WS	Fraction WB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Post-GPC Tray Loc.	Comments
6-21-16	[illegible]	W61855759-1 W61855760-1	30.01	1.0	NA	✓	✓	6-21-16	KF	5mL	6-22-16	JMS	1.0	DD7	2357247-2 SV
		W61855759-2	30.02	1.0		✓						JMS		8	2357247-3 SIM
		-3	30.05				✓							9	
		W61855760-2	29.99				✓							10	
		-3	30.04				✓							C1	
		-4	31.04				✓							2	ms SJ4402-3B
		-5	30.83	✓	✓		✓	✓	✓	✓	✓	✓	✓	3	msd
		GPC Blank #3			6-21-16									4	

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WS	Fraction WB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Post-GPC Tray Loc.	Comments
6-21-16	[illegible]	SJ4402-1E	33.29	1.0	NA	✓		6-21-16	KF	5mL	6-22-16	JMS	1.0	SV12b E5	
		-2E	31.07			✓								6	
		SJ4464-1C	31.12			✓								7	
		-3A	31.16											8	90pm?
		SJ4407-7B	31.10											9	
		-8	30.64											10	
		-9	30.69											SV12b B4	
		SJ4442-3B	30.73				✓							2	msd
		-4B	30.41				✓							3	
		-5A	31.58				✓							4	wet
		-8A	31.31				✓							5	
		-9A	31.66				✓							6	
		-10B	30.39				✓							7	
		-13A	30.52				✓							8	
		-14A	31.01				✓							9	
		SJ4533-26	22.37			✓	✓							10	
		-36	30.47	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	B1	

Reviewed By_____ Date_____

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-551
Revision History
Cover Page
Page 1

TITLE: GRAIN SIZE ANALYSIS

Prepared By: Jessica Quinn Date: 10-1-15

Approved By:

Department Manager: [signature] Date: 10-1-15

Operations Manager: Deborah J. Nadeau Date: 10.1.15

QA Officer: Lisa Dimond Date: 10.01.15

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated title of sections 1.4 + 5.0, Updated method references for NELAC + DoD Added information about hydrometer readings for Sandy samples.	LAD	09/17	09/17
02	Sect. 2 - Updated method summary, Sect. 4 - Added 1/4" Sieve, removed 3/4". Sect. 7 - Updated Sieve only analysis. Updated references and logbook example	LAD	04/19	04/19

TITLE: **Grain Size Analysis**

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TITLE: **Grain Size Analysis**

1.0 SCOPE AND APPLICATION

This SOP details the procedure used by Katahdin Analytical Services technical personnel for particle size analysis in soils. This method is applicable to ASTM D422.

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in Grain Size Analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in Grain Size Analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks

1.3 Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation

from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

For Grain size with hydrometer, depending on total solids of a sample, a certain amount is soaked in a dispersing agent overnight. The sample is then split into two portions, the material retained on the #10 sieve and the material passing the #10 sieve. The material retained on the #10 sieve is dried overnight to a constant weight. The sample is then passed through a large size sieve stack. Material passing the #10 sieve may be subjected to hydrometer analysis. After wet washing the sample on a #200 sieve the sample retained is dried overnight, then passed through a small size sieve stack. The material retained on each sieve, large and small sieves are measured and recorded. All measurements, large and small sieves and hydrometer readings are used to determine the particle size distribution of the sample. If the analysis requires sieve only, the sample weight will be determined as with samples including hydrometer. The samples will then be soaked in DI overnight, than wet washed and baked overnight. They will then be sieved through large and small sieves.

3.0 INTERFERENCES

Not Applicable

4.0 APPARATUS AND MATERIAL

4.1 Sieves ASTM E-11 Specifications, Brand Advantech, of the following size(s):

4.1.1 3.0" (75.00 mm)

4.1.2 2.0” (50.00 mm)

4.1.3 1.5” (37.50 mm)

4.1.4 1.0” (25.00 mm)

4.1.5 3/4" (19.00 mm)

4.1.6 1/4 “ (6.3mm)

4.1.7 # 4 (4.75 mm)

4.1.8 #10 (2.00 mm)

4.1.9 #20 (850.0 um)

4.1.10 #40 (425 um)

4.1.11 #60 (250.0 um)

4.1.12 #80 (180 um)

4.1.13 #100 (150.0 um)

4.1.14 #200 (75.0 um)

4.2 Sedimentation Classico Cylinder(s) 1000 mL

4.3 Hydrometer: ASTM 151H Humboldt H-4242

4.4 Drying Oven with temperature range of 60-110°C

4.5 Stainless Steel Spatulas & Spoons

4.6 Metal & Bristle Brushes

4.7 Ro-Tap Sieve Shaker- Gilson Company

4.8 Timers- capable of counting up to 24hours

4.9 Balance, capable of weight measurement to 0.01 g

4.10 Mechanical Stirring Device and Dispersion Cup- Hamiliton Beach Humboldt

4.11 Thermometer: Accurate to 0.5°C

4.12 Mortar and Rubber Tipped Pestle

4.13 Glass beakers- 1000ml and 500ml

5.0 REAGENTS AND STANDARDS

5.1 Laboratory Grade Reagent Water

5.2 Sodium Hexametaphosphate:

5.3 **Sodium Hexametaphosphate Solution**: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon plastic jug with cover. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 The following information must be recorded in the Grainsize logbook (all that are applicable).

- Start/End Date and Time
- Date/Time placement of samples in and out of oven
- Hydrometer(s) Serial Number
- Hydromter(s) Calibration Date
- Balance ID(s)
- Reagant ID
- Initial and final weights
- Analysts Initials
- Any comments regarding the sample extraction

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

7.2 Equipment Calibration

Calibrate the balances being used each day prior to use. Record in the logbook designated for this purpose.

Calibrate or replace the hydrometers every five years

7.3 Total Solids Determination

Refer to SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”

7.4 Sample Preparation

7.4.1 From the calculated percent total solids and the sample characterisitics for each sample the amount needed for the analysis can be determined using Table 1.

7.4.2 After determining the amount of sample to be used, place a 1000ml glass beaker on the balance and tare the balance. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.4.3 Add 125ml sodium hexametaphosphate solution to each beaker containing sample. Stir and mix thoroughly, soak sample in solution for at least 16 hours.

7.4.4 Refer to Katahdin SOP CA-108, current revision, “Basic Laboratory Technique” for more information on subsampling.

7.5 Sample Partition

After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.5.1 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.5.2 After the sample slurry has been blended, pour sample through a #10 sieve with pan attached, so sample smaller than a #10 sieve is collected in pan. Transfer contents in pan to a 1000ml graduated cylinder and continue to rinse the dispersion cup through the #10 sieve until transfer is complete. After all sample in the pan is rinsed into the cylinder bring the volume of the cylinder to 1000ml using DI water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature for the hydrometer analysis.

7.5.3 Weigh and label a medium aluminum pan, than transfer the contents of the material retained on the # 10 sieve to the pan. Place the aluminum pan in a drying oven set at 110 $\pm$ 5° C and dry the sample material for at least 16 hours or until constant weight set aside for sieve analysis.

7.6 Hydrometer

Prepare a hydrometer blank by adding 125ml sodium hexametaphosphate and bring to 1000 mL with DI water in a 1000 mL graduated cylinder. Be sure to take readings with a hydrometer and a thermometer while taking readings on actual hydrometer samples. This will provide us with the temperature and solution correction factors later in the procedure. Also prepare a hydrometer rinse bath, used to rinse the hydrometer between uses.

7.6.1 To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

7.6.2 To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait approximately 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the logbook. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

7.6.3 Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the logbook. Rinse the temperature probe in the hydrometer rinse bath.

7.6.4 Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes, proceed to small sieve analysis.

With samples that are mostly sand, the hydrometer reading can drop below the reading of the blank. When this occurs, we will apply the lowest hydrometer reading to the blank reading. We will include this in the technical narrative. The logic behind this issue is due to the high amount of sand, resulting in more density. As a result, less DI water is added when transferring the sample/hexametaphosphate solution to the 1000 mL cylinders. Since the blank has a higher DI water/hexametaphosphate ratio, it increases the reading.

7.7 Sieve Analysis for Large and Small Sieves

Look at the sample material in the aluminum pan and record a description of the non-soil material (such as- sticks, grass, wood, plastic), hardness of material and shape of material in the logbook.

Hardness qualifiers include hard, soft or brittle.
Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

7.7.1 Weigh the 3/4", 1/4", #4 and #10 sieves and enter the weight measurements in the logbook as the tare weight.

7.7.2 Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

Small Sieves

Completely transfer the sample from the graduated cylinder to a #200 wet wash sieve. Make sure the entire sample has been transferred to the #200 wet wash sieve by rinsing the graduated cylinder several time with DI water. Using DI water, wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve into a aluminum tin labeled with the sample's LAB ID.

7.7.3 Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

7.7.4 Samples are sieved through the #20, #40, #60, #80, #100, and #200.

7.7.5 After samples have cooled, stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 4 minutes. Weigh each sieve and record these measurements in the logbook.

7.8 Sieve Only Analysis

7.8.1 Determine the total solids of the sample. Use the Percent Solid Table (Table 1) to determine the sample size to be used. If total solids are not available depending on sample matrix we make an educated guess as to how much sample will be used. Samples are then soaked in DI overnight.

7.8.2 After sample and solution has soaked for a minimum of 16 hours, the sample slurry is rinsed into a dispersion cup using DI water. Fill the dispersion cup ½ full with DI water and place the cup on the blender to mix for one minute.

7.8.3 If the sample appears to have large gravel, sand, or organic material that does not appear to be amendable for using the blender notify the PM and proceed to the next step without blending.

7.8.4 Samples are then wet-washed through the #200 Sieve until samples run clear.

7.8.5 Samples are then completely transferred to a metal tin labeled with appropriate sample ID, and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the tin from the oven and allow it to cool.

7.8.6 Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

7.8.7 For Large Sieve analysis follow 7.7.1-7.7.2

7.8.8 For Small Sieve analysis follow sections 7.7.2-7.7.3

7.9 Calculations

7.9.1 Sample Used (SU): total dry sample

SU =Total Sample Weight ((100-%Moisture)/100)*

HMCF = Hygroscopic moisture correction factor (we assume 1)

TITLE: **Grain Size Analysis**

7.9.2 Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

7.9.3 Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1)
Time, minutes = Time of hydrometer reading from beginning of edimentation
Sqrt - square root
SG - Specific Gravity of soil (assuming a default SG)
Viscosity - is the resistance of a liquid to flow

Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))

Intercept = (low temp. reading -1) - (low temp. * slope)

TITLE: **Grain Size Analysis**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not Applicable

9.0 METHOD PERFORMANCE

Not Applicable

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM Standard D 422-63 (Re-approved 2007) “Standard Test Method for Particle-Size Analysis of Soils”, ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

Katahdin SOP SOP CA-717 “Total Solids/Total Volatile Solids Determination In Solid Matrices”, current revision.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1.1, 2018

LIST OF TABLES AND FIGURES

TITLE: **Grain Size Analysis**

TABLE 1

PERCENT SOLIDS TABLE FOR WEIGHT DETERMINATION

Percent Solid Table
Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	50	75	100	200
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

TITLE: **Grain Size Analysis**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-551-02	METHOD ASTM D 422-63
Procedures		
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Accuracy/ Precision		

TITLE: **Grain Size Analysis**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services, LLC.
Sediment Grain Size - Method ASTM D422

Lab Sample ID	SM2456-4	Start Date/Time	3-20-19: 11:51
Analyst:	JMS	End Date/Time	4-2-19: 12:44
Sample Description:	Muck/organics - removed 35.1g sticks/grass/roots		
Sample Weight	Sample (g)	Date/Time in oven	See pg. 1
Sample Weight (wet)	~~125.0~~ 89.9	Date/Time out of oven	〃
Sample Weight (dried)	25.98		

% Moisture	71.105

Sample Split (Oven Dried)	Sample (g)
Sample >=#10	0
Sample <=#10	25.98

Sieve Only:	
Sieve Only, Wet Wash:	

Hydrometer Data	
Serial Number	152303
Cal Date:	3/25/19: 11:32
Low Temp C	16.4
Low Temp Reading	1.0035
High Temp	17.6
High Temp Reading	1.0035
Soil Gravity	2.65

Gravel/Sand Fraction (Sieves)			
Sample Fraction	Size (um)	Pan Tare	Pan+Sample
3"	75000		
2"	50000		
1.5"	37500		
1"	25000		
3/4"	19000		
1/4"	6300		
#4	4750		
#10	2000		
#20	850	307.2	307.5
#40	425	276.0	270.16
#60	250	248.5	249.0
#80	180	241.9	242.1
#100	150	236.5	236.7
#200	75	315.2	315.8
Pan	Pan	339.9	340.43 Dry

11:45	Silt/Clay Fraction (Hydrometer Test)			
Time (min)	Proposed Read Time	Actual Time (min)	Temp C	Spec. Gravity
2	11:50	11:50 (2)	17.2	1.0155
5	11:53	11:53 (5)	17.0	1.0130
15	12:03	12:03 (15)	17.5	1.0115
30	12:18	12:17 (29)	17.43	1.0105
60	12:48	12:48 (60)	16.6	1.0100
240	15:48	15:48 (240)	16.5	1.0075
1440	11:48	11:44 (1441)	17.5	1.0060

EX-028 – Revision 3 – 03/19/2019

QAEX379

0000002

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Hexham Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Figures 2 and 3. Changed KAS, INC. to KAS.	LAD	06/15	06/15
07	Sect. 1 - Replaced beaker with digestion vessel. Sect. 7.5 & 7.9 - Added calibrated pipet.	LAD	06/16	06/16
08	Update figure 1. Change title of section 5.0 to Reagents and Standards. Update method references for NELAC and DoD.	LAD	09/17	09/17
09	Added Thorium to Tables 2 and 3. Sect. 8 - Added contingency plan.	LAD	01/19	01/19

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

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KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever digestion vessels are being heated in the hood. Use caution when handling hot digestion vessels.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95^{o}C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

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4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

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Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter or calibrated pipet, to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter or calibrated pipet to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final

volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-09	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

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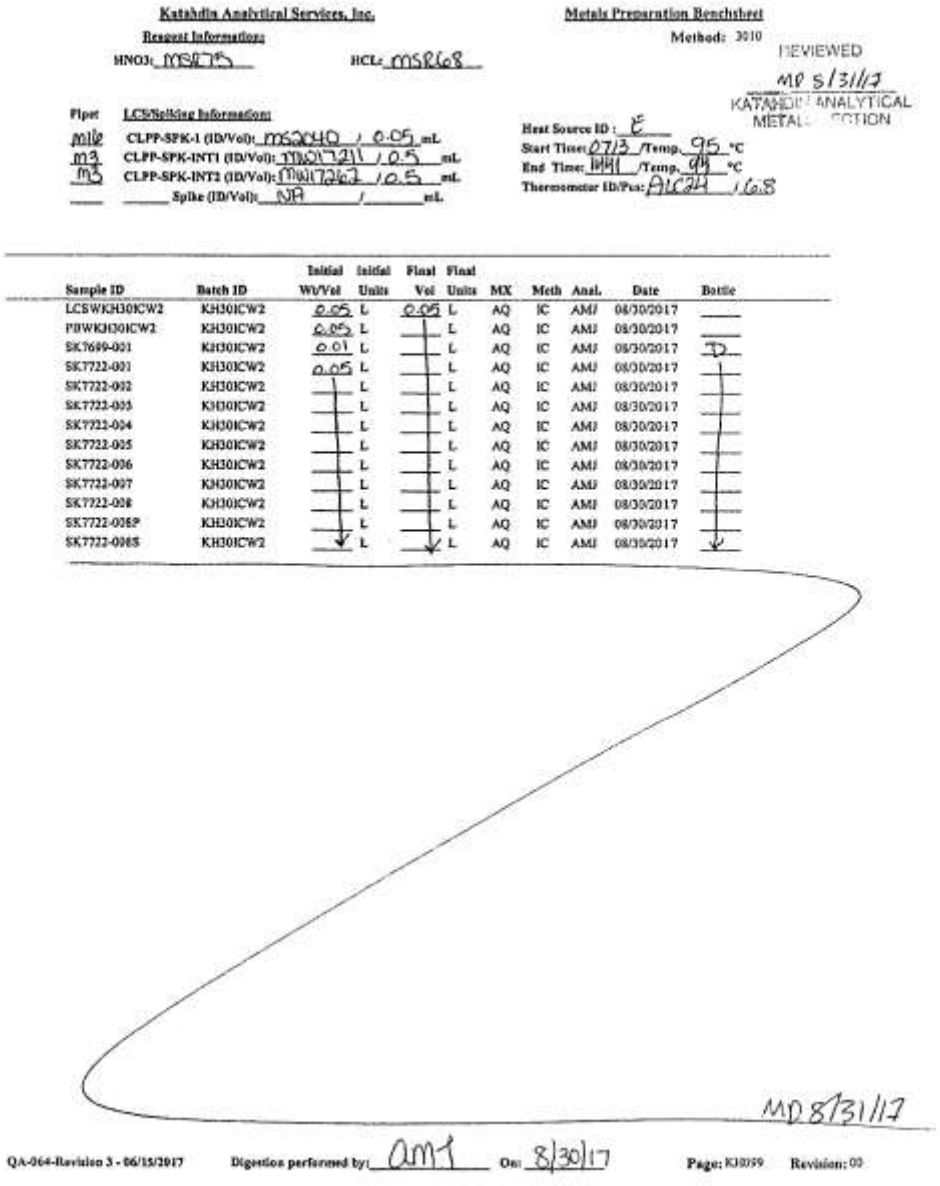
FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:

HNO3: MSB75 HCL: MSB68

Method: 3010

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MP 8/31/17
KATAHDIN ANALYTICAL
METALS SECTION

Pipet LCS/Spiking Information:

m1b CLPP-SPK-1 (ID/Vol): MS2040 / 0.05 mL
m3 CLPP-SPK-INT1 (ID/Vol): MW17211 / 0.5 mL
m5 CLPP-SPK-INT2 (ID/Vol): MW17267 / 0.5 mL
___ _____ Spike (ID/Vol): NA / _____ mL

Heat Source ID: E
Start Time: 0713 /Temp. 95 °C
End Time: 1111 /Temp. 94 °C
Thermometer ID/Pos: A1C24 / 6.8

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWKH30ICW2	KH30ICW2	0.05	L	0.05	L	AQ	IC	AMJ	08/30/2017	
PBWKH30ICW2	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7689-001	KH30ICW2	0.01	L		L	AQ	IC	AMJ	08/30/2017	D
SK7722-001	KH30ICW2	0.05	L		L	AQ	IC	AMJ	08/30/2017	
SK7722-002	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-003	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-004	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-005	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-006	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-007	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008P	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	
SK7722-008S	KH30ICW2		L		L	AQ	IC	AMJ	08/30/2017	

MP 8/31/17

QA-064-Revision 3 - 06/15/2017 Digestion performed by: AMJ On: 8/30/17 Page: KH059 Revision: 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10,000 mg/L K	High Purity Standards	10.0
	10,000 mg/L Na	High Purity Standards	7.5
	10,000 mg/L Mg	High Purity Standards	5.0
	10,000 mg/L Ca	High Purity Standards	2.5
	1000 mg/L Tl	High Purity Standards	1.0
CLPP-SPK-INT2	1000mg/L Sr	High Purity Standards	5.0
	1000mg/L Sn	High Purity Standards	5.0
	10,000mg/L Si	High Purity Standards	1.0
	1000mg/L B	High Purity Standards	5.0
	1000mg/L Li	High Purity Standards	5.0
	1000mg/L Ti	High Purity Standards	5.0
	1000mg/L Mo	High Purity Standards	1.0
	1000mg/L U	High Purity Standards	1.0
	1000mg/L W	High Purity Standards	1.0
	1000mg/L Th	High Purity Standards	1.0

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FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION		
	CLPP-SPK-1	CLPP-SPK-INT1	CLPP-SPK-INT2
Aluminum	2000		
Antimony		10	
Arsenic		10	
Barium	2000		
Beryllium	50		
Boron			50
Cadmium		25	
Calcium		250	
Chromium	200		
Cobalt	500		
Copper	250		
Iron	1000		
Lead		10	
Magnesium		500	
Manganese	500		
Molybdenum			10
Nickel	500		
Potassium		1000	
Selenium		10	
Silicon			100
Silver	50		
Sodium		750	
Strontium			50
Thallium		10	
Tin			50
Titanium			50
Uranium			10
Vanadium	500		
Zinc	500		
Lithium			50
Tungsten			10
Thorium			10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Joel C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Hayden Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DH	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DH	8.29.02	8.29.02
03 3050B	New Title to include ICP-MS. 3. Use of digestion block and polyethylene digestion tubes added to sections 9.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 5.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 7 – Added wording concerning subsampling. Table 3 and 4 – Corrected standard concentrations. Attachment A - Modifications For 8330B Preparation & Digestion. Changed KAS INC. to KAS throughout	UAD	08/15	08/15
07	Update Figure 2. Change title of Section 5.0. Update method references for NELAC + DoD. Minor changes to Table 1 + Section 8.2.	UAD	09/17	09/17
08	Updated Table 3 to include Thallium and corrected standard concentration of U + W. Updated Table 4 to include Thallium Sect. 8 - added Contingency Plan. Sect 10 - updated references	UAD	01/19	01/19

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

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TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSO – Laboratory Control Sample for Solids – An aqueous standard that had been brought through the sample preparation process.

LCSS – Laboratory Control Sample for Solids – A solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with

the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from the Environmental Health and Safety Officer, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature

of each hot plate used is measured and recorded each day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated ($4^oC \pm 2^oC$) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Avoid rocks, roots, leaves and other organic or inorganic foreign material. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet.

Refer to Katahdin Analytical Services SOP CA-108, current revision "Basic Laboratory Technique" for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95^{o}C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^{o}C ± 5^{o}C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 Prepare an appropriate number of laboratory control samples (LCSO for aqueous LCS or LCSS for solid LCS reference material) by weighing appropriate masses of solid reference material or by spiking the LCSO as described in Table 3. The analyte concentrations of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

LIST OF TABLES AND FIGURES

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Aqueous or Solids (LCSO or LCSS)	One each per prep batch of 20 or fewer samples, if specified by project or client	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-08	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	1.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	1.0
	1000 mg/L U	High Purity Standards	1.0
	1000 mg/L W	High Purity Standards	1.0
	1000 mg/L Th	High Purity Standards	1.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L			
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**
Aluminum	2.000	2000		
Antimony	0.100		10	
Arsenic	0.100		10	
Barium	2.000	2000		
Beryllium	0.050	50		
Boron	0.500			50
Cadmium	0.250		25	
Calcium	2.500		250	
Chromium	0.200	200		
Cobalt	0.500	500		
Copper	0.250	250		
Iron	1.000	1000		
Lead	0.100		10	
Lithium	0.500			50
Magnesium	5.000		500	
Manganese	0.500	500		
Molybdenum	0.300			10
Nickel	0.500	500		
Potassium	10.000		1000	
Selenium	0.100		10	
Silicon	5.000			100
Silver	0.050	50		
Sodium	7.500		750	
Strontium	0.500			50
Thallium	0.100		10	
Tin	0.500			50
Titanium	0.500			50
Tungsten	0.100			10
Uranium	0.100			10
Vanadium	0.500	500		
Zinc	0.500	500		
Thorium	0.100			10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet — Method: 3050B

Reagent Information:

HNO3: MSR75 HCL: MSR68 H2O2: MSR62 Filter Paper: 9782497/9831914

Boiling Stones: MSR53

Plate	LCS	Spike	LCS/Spiking Information:
MLD	☒	☒	CLPP-SPK-1 (ID/Vol): MS20640 / 0.1 mL
M3	☒	☒	CLPP-SPK-INT1 (ID/Vol): MW17211 / 1.0 mL
M3	☒	☒	CLPP-SPK-INT2 (ID/Vol): MW17262 / 1.0 mL
—	☒	☒	Spike (ID/Vol): NA / mL

LCSS: NA Balance ID: Bal15

Heat Source ID: E

Start Time: 0749 /Temp. 91 °C

End Time: 1421 /Temp. 94 °C

Thermometer ID/Pos: AIC24 / 4.1

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KATAHDIN ANALYTICAL
METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKJ01ICS1	KJ01ICS1	1.0	g	0.1	L	SL	IC	AMJ	09/01/2017	—
PBSKJ01ICS1	KJ01ICS1	1.0	g		L	SL	IC	AMJ	09/01/2017	—
SK7769-001	KJ01ICS1	1.88	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001P	KJ01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7769-001S	KJ01ICS1	1.89	g		L	SL	IC	AMJ	09/01/2017	A
SK7789-001	KJ01ICS1	1.20	g		L	SL	IC	AMJ	09/01/2017	G
SK7794-001	KJ01ICS1	1.95	g		L	SL	IC	AMJ	09/01/2017	A
SK7794-002	KJ01ICS1	1.98	g		L	SL	IC	AMJ	09/01/2017	〃
SK7794-003	KJ01ICS1	1.92	g		L	SL	IC	AMJ	09/01/2017	〃
SK7837-001	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-002	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-003	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-004	KJ01ICS1	0.11	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-005	KJ01ICS1	1.0	g		L	AR	IC	AMJ	09/01/2017	〃
SK7837-006	KJ01ICS1	1.0	g	↓	L	AR	IC	AMJ	09/01/2017	↓

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TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 158
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

ATTACHMENT 1

MODIFICATIONS FOR 8330B PREPARATION & DIGESTION

4.0 APPARATUS AND MATERIALS – additional materials

4.1 Sieves - #10 mesh (2 mm) – 8" diameter with covers and collection trays.

4.2 Aluminum drying trays with drying rack

4.3 Heavy duty aluminum foil

4.4 Stainless steel scoopulas

4.5 Dust mask

9.0 PROCEDURES – additional procedures

Prior to the digestion of samples (section 7.1 in SOP):

Spread the entire aliquot of soil onto a drying tray lined with heavy duty aluminum foil and dry in air at room temperature or colder to a constant weight (last two successive dry weights within 3% RPD). Trays should be placed in rack for drying. Record all weights in the Sample Drying Logbook.

Note: Hydric soils and sediments with high moisture content may take several days to dry to constant weight.

Remove the oversize fraction by passing it through a 10-mesh (2 mm) sieve. Be sure to break up caked up soil with a gloved hand. Weigh both fractions – oversize and <2mm. Record all weights in the Sieving & Grinding Logbook.

To obtain a subsample, the entire sample must be mixed with a stainless steel scoopula and spread out on a clean surface (aluminum tray lined with foil) so that it is only 1 or 2 cm thick - preferably in a fume hood designed to prevent the spread of dust and possible inhalation or residue losses. Using the scoopula, obtain at least 30 different increments, i.e., portions (~0.3 g) from randomly chosen locations throughout the entire sample profile for a total of ~10 g. Mix this subsample one more time with the scapula and then obtain an aliquot for metals digestion (beginning with section 7.3).

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-608
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Bunton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Duncan F. Hufnagle Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1·23·01	1/23/01
02 6010B	Calibration begins with analysis of S0 (Cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element conc's. in Tables 3, 4, 5, 6 to reflect current practices.	DH	10·21·02	10·21·02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current Standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated Sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up Updated tables to reflect changes in Standard Concentrations and Preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for Compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10
12	Revised Tables 4 → 8 with the following information: -Add palladium and gold; removed tungsten and uranium; removed Stock Standard ~~ICP-CICV~~ 200.7ICS-1; changed stock standard ICP-CICV-3 to CL-CAL-3. Added references to section 10.	LAD 09/22/11 LAD	09/11	09/11
13	The changes above had not been finalized in SOP-12. Sect. 9 - Added MDL, LOD and LOQ information. Added Attachment 2 - Analysis of palladium by SW846 6010	LAD	04/12	04/12

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 9 & Table 1 - Fixed typos.	LAD	05/13	05/13
15	Sect. 10 - Updated references. Added Table 3 - DoDQSM 5.0 QC Requirements - Renumbered rest of Tables. Updated Tables (6→8). Changed KAS INC to KAS LLC.	LAD	12/14	12/14
16	Sect. 5 & 7 - corrected Table references. Tables 5, 6, 7 & 8 - Updated Standard, Concentrations & sources. changed KAS LLC to KAS	LAD	05/16	05/16
17	Sect. 1 and 6 - Added Tissue matrix	LAD	07/16	07/16
18	Sect. 8.1 - changed reagent spiked water to calibration blank solution. Sect. 10 - update method references.	LAD	09/17	09/17
19	Updated Tables to correct concentrations for a few elements. Removed Table 2, DoDQSM 4.2 QC Requirements. Renumbered Tables	LAD	01/19	01/19

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-19**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-19**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, LLC personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, biological tissue and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution

ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document

their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual l and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of

the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 4 and 5 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months
Tissue	P, G	25 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multi-element calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Tables 1 through 3 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Tables 1 through 3 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and

project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed. Tables 2 and 3 list the QC Check, minimum frequencies, acceptance criteria, corrective actions, flagging criteria and additional comments for work analyzed in accordance with DoD QSM versions 4.2 and 5.0.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of calibration blank solution, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements

may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within $\pm$ PQL if the PQL is greater than 0.01 mg/L, within $\pm$ 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run,

unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM 4.2 acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. If DoD QSM 5.0 acceptance criteria are being used, recovery for water and solid matrix samples must fall between the limits stated in Tables 3 & 4 of the QSM. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in

all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 6010C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

List of Tables & Figures

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the LLCCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike(MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-18	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 6)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	Calibration Standard 3	Claritas PPT	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	Inorganic Ventures	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn, Au	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 6)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 6)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 6)	1.0

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TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L Li, Sn, Au	High Purity Standards	1.0 each
	10000 mg/L K, Na	High Purity Standards	1.0 each
	1000 mg/l B	High Purity Standards	0.50
	1000 mg/l Zn	High Purity Standards	0.20
	1000 mg/L Cu	High Purity Standards	0.25
	10000 mg/L Si	High Purity Standards	0.20
	1000 mg/L Ti, Tl	High Purity Standards	0.15 each
	1000 mg/L Se, Mo, Co, Ni, Ag, Sr, V, Cr	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba, Be, Cd, Mn, Pb	High Purity Standards	0.05 each
	10000 mg/L Fe, Ca, Mg	High Purity Standards	0.1 each
ICSAB-INT	1000 mg/L K,Na	High Purity Standards	4.0 each
	1000 mg/L B, Li, Mo,Sr,Sn,Ti, Au	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.40
ICP-INT STD (Intermediate)	10000 mg/L Si	High Purity Standards	2.5
	10000 mg/L Ca, Mg, Fe, Al, Na	High Purity Standards	2.4
	10000 mg/L K	High Purity Standards	1.5
	1000 mg/L Au, Li, Sn. Sr	High Purity Standards	1.0

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TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L							
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**
Aluminum	25	10	0.3	500	500	12.5	500	
Antimony	1	0.4	0.008		0.6	0.5		
Arsenic	1	0.4	0.008		0.1	0.5		
Barium	1	0.4	0.005		0.5	0.5		
Beryllium	1	0.4	0.005		0.5	0.5		
Boron	1	0.4	0.05		0.5	0.5		
Cadmium	1	0.4	0.005		1.0	0.5		
Calcium	25	10	0.10	500	500	12.5		
Chromium	1	0.4	0.01		0.5	0.5		
Cobalt	1	0.4	0.01		0.5	0.5		
Copper	1	0.4	0.025		0.5	0.5		
Iron	25	10	0.1	200	200	12.5		200
Lead	1	0.4	0.005		0.05	0.5		
Lithium	1	0.4	0.1		0.5	0.5		
Magnesium	25	10	0.10	500	500	12.5		
Manganese	1	0.4	0.005		0.5	0.5		
Molybdenum	1	0.4	0.01		0.5	0.5		
Nickel	1	0.4	0.01		1.0	0.5		
Potassium	25	13.6	1		20	12.5		
Selenium	1	0.4	0.01		0.05	0.5		
Silicon	25.5	10.0	0.2		2	12.75		
Silver	1	0.4	0.01		0.2	0.5		
Sodium	25	10	1		20	12.5		
Strontium	1	0.4	0.01		0.5	0.5		
Thallium	1	0.4	0.015		0.1	0.5		
Tin	1	0.4	0.1		0.5	0.5		
Titanium	1	0.4	0.015		0.5	0.5		
Vanadium	1	0.4	0.01		0.5	0.5		
Zinc	1	0.4	0.02		1.0	0.5		
Gold	1	0.4	0.1		0.5	0.5		

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TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP Intermed STD	PQL-INT	ICSAB-INT
Aluminum	240	30	
Antimony		0.8	
Arsenic		0.8	
Barium		0.5	
Beryllium		0.5	
Boron		5	10
Cadmium		0.5	
Calcium	240	10	
Chromium		1.0	
Cobalt		1.0	
Copper		2.5	
Iron	240	10	
Lead		0.5	
Lithium	10	10	10
Magnesium	240	10	
Manganese		0.5	
Molybdenum		1.0	10
Nickel		1.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.0	
Sodium	240	100	400
Strontium	10	1.0	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	10
Vanadium		1.0	
Zinc		2.0	
Gold	10	10	10

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TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	IV-28	QCS-26	CLPP-ICS-A	CLPP-ICS-B4	CL-CAL-3
Aluminum	100	100	5000		
Antimony	100	100		60	
Arsenic	100	100		10	
Barium	100	100		50	
Beryllium	100	100		50	
Boron	100	100			
Cadmium	100	100		100	
Calcium	100	100	5000		1000
Chromium	100	100		50	
Cobalt	100	100		50	
Copper	100	100		50	
Iron	100	100	2000		1000
Lead	100	100		5	
Lithium	100				
Magnesium	100	100	5000		1000
Manganese	100	100		50	
Molybdenum	100	100			
Nickel	100	100		100	
Potassium	1000	1000			1000
Selenium	100	100		5	
Silicon	50	50			
Silver	100	100		20	
Sodium	100	100			1000
Strontium	100				
Thallium	100	100		10	
Tin					
Titanium	100	100			
Vanadium	100	100		50	
Zinc	100	100		100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 2

ANALYSIS OF PALLADIUM BY SW846 6010

Palladium may be analyzed by EPA Method SW846 6010C following the method outlined in this SOP. However, due to significant spectral interferences caused by addition of palladium to the calibration and check standards used in this method, palladium is added to aliquots of the regular standards as needed for analysis. Two stock standards (1000 mg/L) are currently kept for palladium analysis. One is purchased from High Purity Standards and is used for calibration, PQL, ICSAB, and CCV. The other is purchased from Inorganic Ventures and is used as the independent check standard (ICV). Analysts should add palladium stock to the regular standards according to the table below:

Name of Working Standard	Volume of Standard Aliquot (mL)	Volume of Palladium Stock Added (mL)	Concentration of Palladium (mg/L)	Source of Palladium Stock
Calibration Std.	50	0.05	1.0	High Purity
ICV	50	0.02	0.4	Inorganic Ventures
PQL	50	0.005	0.1	High Purity
ICSAB	50	0.025	0.5	High Purity
CCV	50	0.025	0.5	High Purity

Prior to starting the run, a palladium-only standard should be analyzed along with the iron and aluminum standards to evaluate interelement correction factors as outlined in Katahdin SOP CA-632, Section 7.1.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Busta Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Murphy Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	DN	1.29.01	1/29/01
03 7471A	Changed LECMAN PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised Sect. 10 to show correct reference materials. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (LCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect. 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DN	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10
08	Sect. 4.6 - Changed thermometer type. Added LCS0 - A LCS prepped using aqueous mercury LCSspike. Updated type of marker used to label digestion bottles. Updated corrective action for failing PQL standard.	LAD	12/10	12/10
09	Sect. 7 - Changed calibration digestion from digestion of all points to digestion of high point and dilutions of rest. Changed prep from 3 to 0.2g aliquots to 1 ~ 0.6g aliquot. Added additional prep info. Added Serial dilution and PDS to sect. 8, Added MDL, LOD, LOQ info. to sect. 9. Updated and added references to Sect. 10.	LAD	04/12	04/12
10	Sect. 7 - Corrected Calibration preparation, changed digestion temperature to 95 +/- 3°C. Sect. 10 - Added and updated references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 4 - Added snap-top containers and digestion tubes. Sect 5 - Updated Aqua Regia prep. Sect. 7 - Added heat block and digestion tube instructions, minor edits. Removed Table 2, updated fig. 1, added fig. 4 Changed ~~KASINC~~ KAS throughout. KASINC to LAD 102517	LAD	10/17	10/17
12	Sect. 7 - Updated to reflect current Calibration and Independent Calibration verification Standard preparation. Corrected typos	LAD	01/19	01/19
13	Sect. 7 - ~~Corrected~~ Corrected Calibration high Standard and ICV preparation. Sect 8 - Added if CCV or LCS fail high and samples ND - report and narrate	LAD	10/19	10/19

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-13, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-13, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

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Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, sludges and tissue under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process. LCSS utilizes the standard reference material. LCSO is spiked with aqueous mercury LCS spike.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps or digestion tubes, for use as digestion vessels.

4.2 Heat source capable of maintaining a constant temperature of 95°C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Thermometer, NIST-traceable, covering the range from -10^{o} to 110^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metal's laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

4.9 4oz graduated snap-cap container, 120 mL capacity.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 1:1 Aqua regia: Prepare an appropriate amount immediately before use. Start with 4 parts laboratory reagent grade water, carefully add one part of concentrated HNO_3 and then three parts of concentrated HCl in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood. Record preparation in "Metals Preparation Laboratory - Reagent Preparation Logbook". Refer to Figure 4 for a cop of a page from this logbook

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook

maintained in the Section. Intermediate Mercury Standard A must be prepared monthly and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared monthly and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Glass mercury digestion bottles are reused and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles

are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights and final volumes, pertinent observations, etc.) must be recorded on this bench sheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using an industrial marker with super permanent ink, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control sample and matrix spike sample to be digested.

7.6 Calibration Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to two standard digestion bottles (250 mL media bottles). Using a dose cup, add 10 mL 1:1Aqua Regia. Using a calibrated adjustable pipette prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to the appropriate media bottle. Using a clean dose cup add 15 mL potassium permanganate solution. Using a repipetter, add 6 mL sodium chloride-hydroxylamine and swirl to mix. Fill the calibration standard to 100 mL with laboratory reagent water and allow to stand. The Mercury concentration of this standard is 10.0 ug/L.

The blank calibration standard is prepared in the same manner as the high calibration standard except for the addition of mercury standard.
Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5.0 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount Calibration Blank Solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.7 Initial Calibration Verification Standard (ICV) Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to a standard digestion bottles (250 mL media bottles). Using a dose cup, add 10 mL 1:1 Aqua Regia. Using a calibrated adjustable pipette prepare the high calibration standard by adding 600 uL of Intermediate Mercury Standard B to the appropriate media bottle. Using a clean dose cup add 15 mL potassium permanganate solution. Using a repipetter, add 6 mL sodium chloride-hydroxylamine and swirl to mix. Fill the calibration standard to 100 mL with laboratory reagent water and allow to stand. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS or LCSO) by weighing appropriate masses of solid reference material or by adding 500 uL of Intermediate Mercury Standard A respectively into labeled digestion bottles. The mercury concentration of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material. The mercury concentration of the LCSO will be 5.0 ug/L.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 Preparation blanks, laboratory control spike and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP. Calibration standards are not digested.

SAMPLE PREPARATION AND DIGESTION

7.12 Do not decant any water on the sediment sample. **Note:** Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

Mix sample with a wooden spatula to ensure homogeneity of the sample. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on sub-sampling to ensure reproducibility.

Weigh an approximate 0.6 g portion of untreated, homogenized sample from the sample container and place in the bottom of a labeled digestion bottle.

7.13 Add 10 mL of 1:1 Aqua Regia to each sample, standard, and QC sample. Place bottles in a heat source located in a fume hood and heat for 2 minutes at 95 ±3°C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 For glass mercury bottle preparation, add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For mercury digestion tube preparation, add 20 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95°C. Record initial and final time and temperatures on the mercury preparation bench sheet.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95 ±3 ^{O}c. Record any information regarding additional permanganate aliquots on the mercury preparation bench sheet and accordingly adjust the final volumes recorded on the bench sheet for the samples affected.

7.16 For glass mercury bottle preparation, add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50

mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.
For mercury digestion tube preparation, quantitatively transfer sample to 4oz snap cap and then add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Bring sample to 100 mL final volume with reagent grade water. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer”. The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471 and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in “templates”. The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named “SW846-7470-7471”.

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument’s autosampler. The “standards” autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of the “samples” autosampler racks. The “samples” autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the “samples” autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer”.

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for

a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2. A linear regression program may be used to obtain the intercept concentration.

7.22 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.23 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported – the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{I \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.24 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are

described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte

standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.4 Instrument calibration – The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

Samples that are below the reporting limit may be reported if the CCV reads greater than 110%.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. If the PQL fails, results may not be reported from the run until the problem is corrected and a passing PQL has been analyzed.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSS or LCSO), consisting of solid reference material or 500 Ul of Intermediate Standard A carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples that are supplied by the vendor of the solid reference material. The results of the LCSO must fall within 80% - 120% of its true value which is 5.0 ug/L. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS or LCSO must be within 80% - 120%.

Samples that are below the reporting limit may be reported if the LCSS or LCSO reads greater than 120%.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 Serial Dilution – A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.14 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantiaion (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning of run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration > PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS or LCSO)	One per digestion batch of 20 or fewer samples.	LCSS: Recovery within vendor-supplied acceptance limits. LCSO: Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	1)Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes or duplicate samples.	Flag results
	Post-Digestion Matrix Spike Sample (PDS)	When matrix spike or MSD fail	Recovery ±20% of true value	Analyze serial dilution of sample
	Serial Dilution Test (L)	One per digestion batch or when PDS fails	1:5 dilution of sample must agree within 10% with undiluted result	If MS, MSD, PDS, and serial dilution fail, quantitate sample by method of standard additions
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		not listed, use in-house LCS limits if project limits are not specified.	failed analytes, if sufficient sample material is available.	specific analyte(s) in all samples in the associated preparatory batch.	reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 3

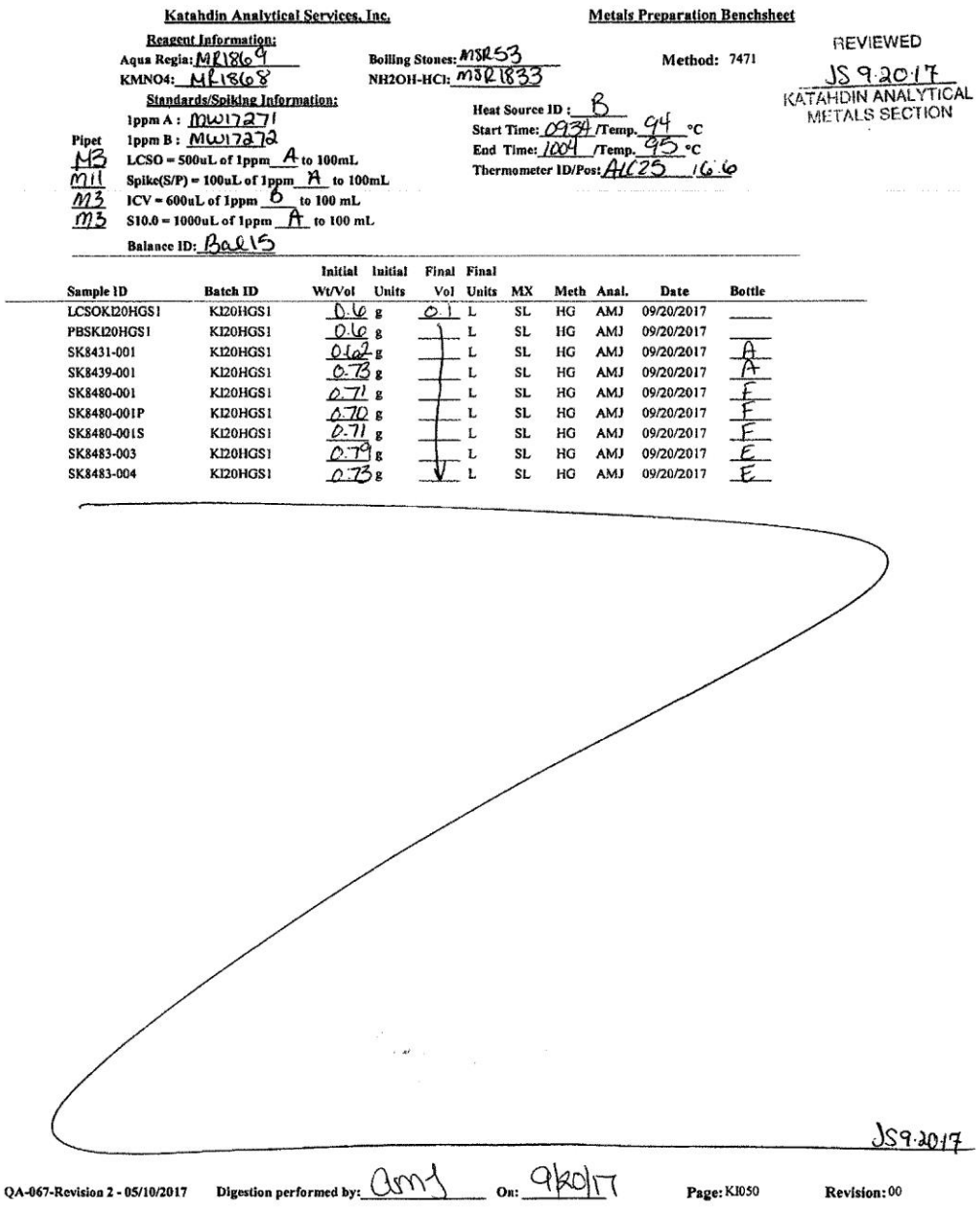
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-12	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
Qc _ Calibration	Calibration standards are not digested.	Sect. 7.3- Requires Calibration standards are digested
QC – Calibration Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
Aqua Regia: MR1869 — Boiling Stones: MSR53 — Method: 7471
KMNO4: MR1868 — NH2OH-HCl: MSR1833

REVIEWED JS 9.20.17 KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:
1ppm A: MW17271
1ppm B: MW17272
Pipet
M3 — LCSO = 500uL of 1ppm A to 100mL
M11 — Spike(S/P) = 100uL of 1ppm A to 100mL
M3 — ICV = 600uL of 1ppm B to 100 mL
M3 — S10.0 = 1000uL of 1ppm A to 100 mL
Balance ID: Bal15

Heat Source ID: B
Start Time: 0934 /Temp. 94 °C
End Time: 1004 /Temp. 95 °C
Thermometer ID/Pos: AHC25 16.6

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOKI20HGS1	KI20HGS1	0.6	g	0.1	L	SL	HG	AMJ	09/20/2017	
PBSKI20HGS1	KI20HGS1	0.6	g		L	SL	HG	AMJ	09/20/2017	
SK8431-001	KI20HGS1	0.62	g		L	SL	HG	AMJ	09/20/2017	A
SK8439-001	KI20HGS1	0.73	g		L	SL	HG	AMJ	09/20/2017	A
SK8480-001	KI20HGS1	0.71	g		L	SL	HG	AMJ	09/20/2017	F
SK8480-001P	KI20HGS1	0.70	g		L	SL	HG	AMJ	09/20/2017	F
SK8480-001S	KI20HGS1	0.71	g		L	SL	HG	AMJ	09/20/2017	F
SK8483-003	KI20HGS1	0.79	g		L	SL	HG	AMJ	09/20/2017	E
SK8483-004	KI20HGS1	0.73	g		L	SL	HG	AMJ	09/20/2017	E

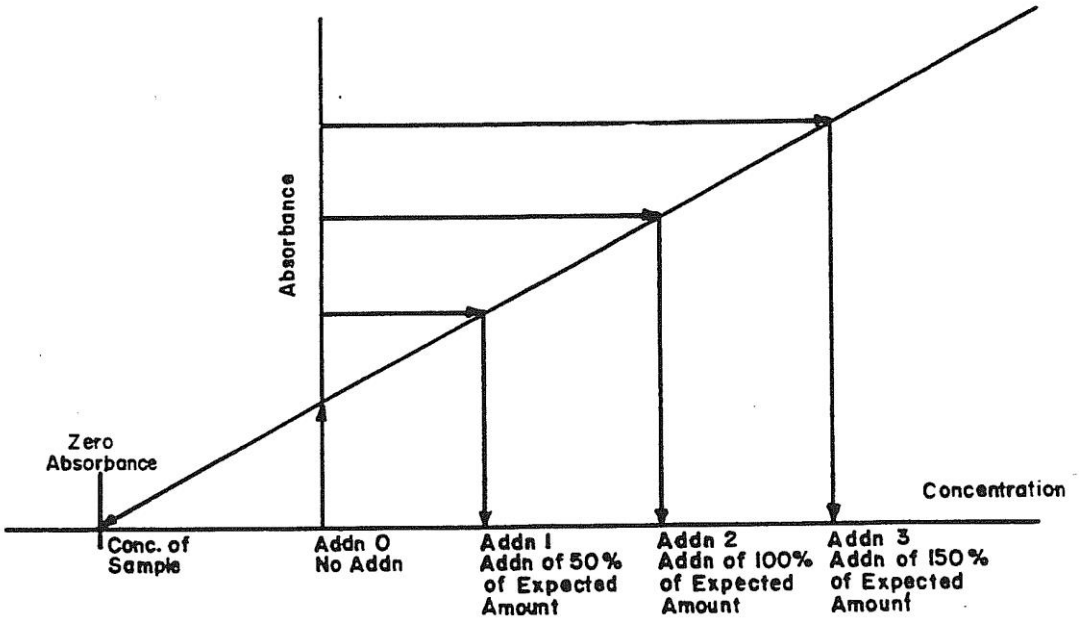
JS 9.2017

QA-067-Revision 2 - 05/10/2017 Digestion performed by: AMJ On: 9/20/17 Page: KI050 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 4

EXAMPLE REAGENT PREP LOGBOOK PAGE

REAGENT PREP LOG - METALS PREP LAB

Reagent ID	Reagent Name	Prep Date	Exp. Date	Prep Initials	Component	Lot Number	Amount Added	Prep Notes
MR1863	$SnCl_2$ - HCL	9-8-17	12-8-17	AMJ	$SnCl_2$	MSR66/MSR76		FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc HCL	MSR68	280mL	↓
MR1864	1:1 Aqua Regia	9-12-17	9-13-17	AMJ	Conc. HCL	MSR68	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR75	50mL	↓
MR1865	NH_2OH - HCL (12% w/v)	9-14-17	AMJ 9/14/17 @ 12-14-17	AMJ	NH_2OH	MSR57/MSR58	480.01	FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	NaCL	MSR36	480.01	↓
MR1866	1:1 aqua Regia	9-18-17	9-19-17	AMJ	Conc. HCL	MSR68	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR77	50mL	↓
MR1867	5% HNO_3	9-18-17	9-18-18	AMJ	Conc. HNO_3	MSR77	500mL	FV= 10L w/ Reag H_2O
MR1868	5% (M/v) $KMNO_4$	9-18-17	AMJ 9/18 9/12-18-17	AMJ	$KMNO_4$	35309	200.00g	FV= ~~4000~~ 4L w/ Reag H_2O AMJ 9/18/17
MR1869	1:1 aqua Regia	9-20-17	9-21-17	AMJ	Conc HCL	MSR73	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR77	50mL	↓

ME-011 - Revision 1 - 07/25/2013

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

ADDENDUM A

TISSUE PREPARATION

Prepare tissue samples in the same manner as soils with the following exceptions.

Tissue Preparation	EPA 7471B
Weigh 0.2 to 0.3 g portions of each sample	Weigh 0.5 to 0.6 g portions of each sample
Add 4 mL on concentrated h2so4 and 1 mL of concentrated HNO3 to each bottle	Add 5 mL H2O and 5 mL of Aqua Regia
Heat to 58°C for 30-60 minutes	Heat at 95°C for 2 minutes
Cool to 4°C in an ice bath	Cool to room temperature
Add 5 mL of potassium permanganate solution in 1 mL increments	Add 50 mL H2O and 15 mL potassium permanganate
Add an additional 10 mL potassium permanganate or more to maintain oxidizing conditions	
Add 8 mL of potassium persulfate	
Allow to stand overnight at room temperature	Heat at 95°C for 30 minutes

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis P. Hogan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1.29.01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02-16-05	02-16-05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoDQSM version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.6 - changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard corrective action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12
08	DoDQSM 5.0 References added. Sect. 7.4 and Table 3 - Updated Calibration Standard prep - removing digesting all Standards. Added to digest high point	LAD	06/14	06/14
09	Updated Figure 1. Change title of section 5.0. Update method references for NELAC and DoD. Minor additions to sections 4.1, 4.2, 4.6, 7.1, 7.10, 7.12.	LAD	09/17	09/17
10	Removed DoDQSM 4.2 QC Requirement Table. Added DoDQSM 5.0/5.1 QC Requirement Table. Updated references. Updated Logbook example	LAD	11/18	11/18
11	Sect. 7 - Updated to reflect current calibration and Independent Calibration Verification Standards	LAD	01/19	01/19

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-11,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of

hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL, 50 mL or 70 mL digestion tubes and appropriate watch glasses, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in preparation of calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50^{o} to 750^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate

Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL, 50 mL or 70 mL digestion tubes. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using an industrial marker with super permanent ink, label clean sample containers with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control spike and matrix spike and standard to be digested.

7.4 Calibration Preparation - Using a bottle-top dispenser, add approximately 10 mL of laboratory grade reagent water to two standard digestion bottles (250 mL media bottles). Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to each bottle. The blank calibration standard is prepared in the same manner as the high calibration standard except for the addition of mercury standard. Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to the appropriately labeled media bottle. Using a dose cup, add 15 mL potassium permanganate to each calibration standard. Using a repipettor, add 8 mL potassium persulfate solution to each calibration standard and swirl to mix. Fill each bottle to 160 mL with laboratory grade reagent water. The mercury concentration of the high calibration standard is 10.0 ug/L.

Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5 ug/L standards are analyzed after calibration as the PQL standard and

the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount calibration blank solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.5 Independent Calibration Verification Standard - Using a bottle-top dispenser add approximately 10 mL of laboratory grade reagent water to a digestion bottle. Using a repipettor, add 2.5 mL concentrated nitric acid and 5 mL concentrated sulfuric acid to the bottle. Using a calibrated adjustable pipette, add 600 uL of Intermediate Mercury Standard B to the bottle. Using a dose cup, add 15 mL potassium permanganate to each calibration standard. Using a repipettor, add 8 mL potassium persulfate solution and swirl to mix. Fill to 160 mL with laboratory grade reagent water. The mercury concentration of the ICV standard is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 Preparation blanks, laboratory control spikes and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13 but the standards are not heated.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup or pour directly into graduated sample tube, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that

contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

Some samples may require dilution to 25 mL with potassium permanganate for digestion to be performed in the digestion vessel. Prepare method blank and LCS with equal amounts of potassium permanganate to check for potential mercury contamination.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials, for 50 mL or 70 mL tubes, add ribbed watch glasses, and place them in a preheated water bath or heat source. Monitor the temperature of the bath with a thermometer throughout the digestion. The temperature of the water bath will fall below 90-95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 90-95^{o} C, continue heating the samples at 90-95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath or heat source and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 90-95^{o} C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. After color change, samples are filled to 40 mL with laboratory grade reagent water and mixed well. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer”.

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METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B)V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is

shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{\text{MC X DF X IV}}{\text{FV}}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a “U” qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard

deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of

Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is

less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.14 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018.

The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.
Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq$ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm$ 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm$ 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm$25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm$ 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Table 2

DOD QSM 5.0/5.1 REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 3

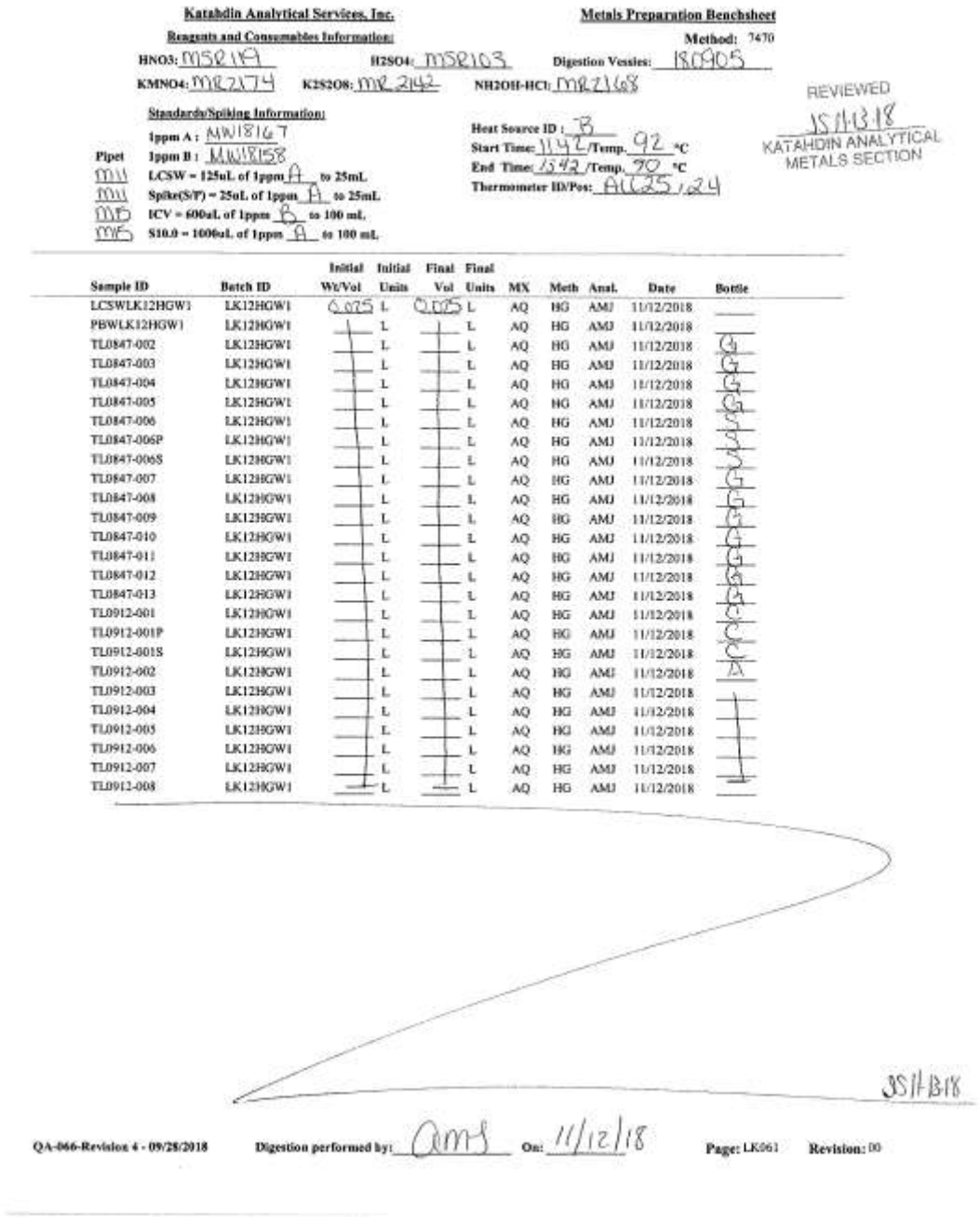
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-11	USEPA METHOD 7470
Reagents	1) Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	1) Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	2) Sampling and gas stream switching performed automatically by mercury analyzer. 3) Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	2) Sampling and gas stream switching performed manually by analyst. 3) Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	4) Known reference sample (ICV) analyzed daily. 5) Calibration verified after every 10 samples with CCV.	4) Known reference sample analyzed quarterly. 5) Calibration verified after every 20 samples.
QC - Calibration Blanks	6) Acceptance criteria employed for 245.1: ± PQL	6) Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagents and Consumables Information: Method: 7470

HNO3: MS0149 H2SO4: MS2103 Digestion Vessles: 180905

KMNO4: MR2174 K2S2O8: MR2142 NH2OH-HCl: MR2168

REVIEWED 1/3/18 KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:

1ppm A : MW18167

1ppm B : MW18158

Pipet

MM1 LCSW = 125uL of 1ppm A to 25mL

MM1 Spike(S/P) = 25uL of 1ppm A to 25mL

MP5 ICV = 600uL of 1ppm B to 100 mL

MM5 S10.0 = 1000uL of 1ppm A to 100 mL

Heat Source ID : B

Start Time: 1142 /Temp. 92 °C

End Time: 1542 /Temp. 90 °C

Thermometer ID/Pos: AC25,24

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWLK12HGW1	LK12HGW1	0.025	L	0.025	L	AQ	HG	AMJ	11/12/2018	
PBWLK12HGW1	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-006S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-009	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-010	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-011	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-012	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-013	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-001S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	

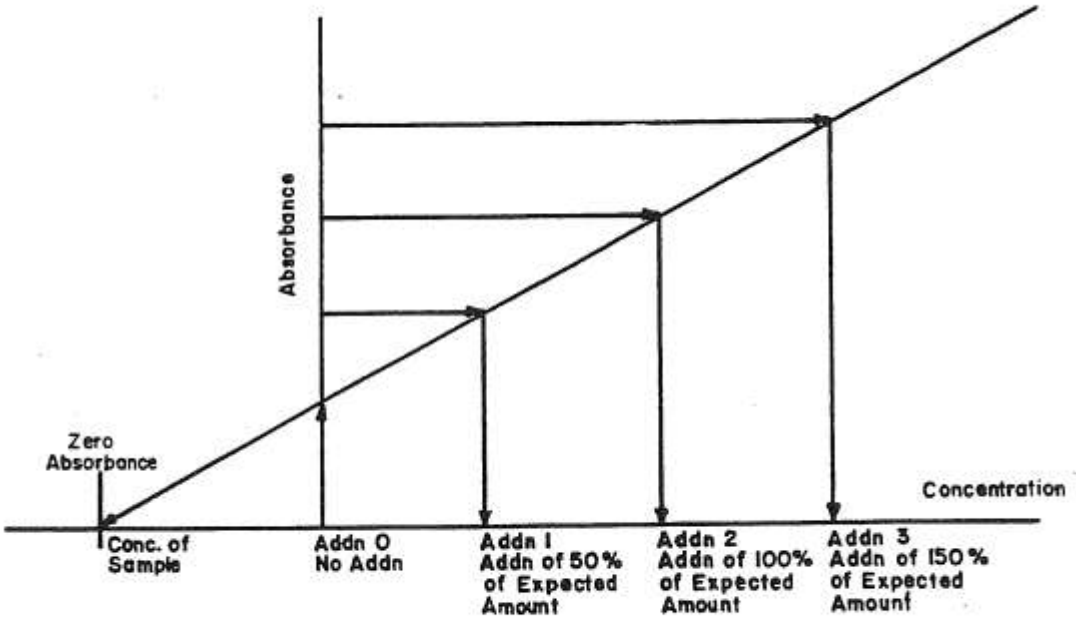
JS 11-13-18

QA-066-Revision 4 - 09/28/2018 Digestion performed by: AMJ On: 11/12/18 Page: LK061 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Jol C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03.27.01

General Manager: Dennis P. Hufford Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. Changed Run ID Naming convention added data reduction and reporting procedures updated Standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tebing size Sect. 5 - changed acid conc.'s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated Tables 1, 3-8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards. Updated Table 1 with Serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1-updated acceptance criteria and corrective action for QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 602A. Added Hardness by calculation attachment. Added LLOC requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 4.1, 4.2, 5.2 7.9, 7.10, 7.11, 7.16 and 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect. 10 - Added, ~~edited references~~. Updated Tables 1, 5, 6, 7, and 8 edited references LAD 042512	LAD	04/12	04/12
09	Sect. 7 - Added reference to autosampler software, added printing calibration and removed printing of run summary	LAD	08/13	08/13
10	Sect. 7 - Updated for changes made in the metals database for importing and handling data. Sect. 10 - updated and added references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 7, Table 1, 2, 3, 6, 8 & 11 - Updated to reflect change from 5 pt. to 2 pt. calibration. Table 7, 8 & 9 - Updated to reflect change in Aluminum PQL	LAD	04/16	04/16
12	Change title of Section 5.0. Update method references for NELAC + DoD. Minor additions/corrections to sections 3.0, 4.2, 7.3.5 and table 5.	LAD	09/17	09/17
13	Table 1 - Added ms/msD, corrected section references - Table 2 - Removed, DoD QSM 4.2 QC Requirements. Renumbered subsequent sections. Table 7 - Added Thorium - Updated Table references throughout SOP	LAD	01/19	01/19

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-13**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-13**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions. If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.
LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) = (m/z 75 signal) - (2.95) (m/z 77 signal) + (2.548) (m/z 82 signal) – (2.571) (m/z 83 signal), where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).

Similarly:
Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.84)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 4. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been

found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See Table 14 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX Fast Autosampler and Sample

Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 3-stop ESI PVC flared black-black (0.76 mm ID) and orange-green-orange (0.38 mm ID). 2-stop ESI PVC flared red-red (1.14 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker “Instra-Analyzed” trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All

acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 6 and 7 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 8 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software. The main Chemstation screen is accessed by double-clicking the “ICP-MS Top” icon on the Windows desktop. Autosampler tables are edited by selecting “Edit Sample Log Table” from the Sequence menu in the Agilent Chemstation software. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 11.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open ESI autosampler software by double-clicking the "ESI SC" icon. Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On** and allow the instrument to aspirate calibration blank solution for at least 45 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 45 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 13.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate the P/A tuning solution (see Table6) and run a P/A auto tune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting:

Methods>>Load>>K1PTCAL16.M>>K1PTCAL16.C.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, select "Edit Sample Log Table" from the **Sequence** menu in the Agilent Chemstation software. Double-click **SMPL** from the menu at the top left. Fill in the sample table with sample IDs, vial numbers, analytical method (K1PTCAL16.M for all samples), dilution factors, and failure actions. When the sample table is complete, select **Print** to print this table. Close the "Edit Sample Log Table" window. Save the sample log table under a new name by selecting **Save** under the **Sequence** menu and then typing the name.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("J" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("G" for 2013, "H" for 2014, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "JGA16A" is the first run of the day that was performed on January 16, 2013, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least one multielement calibration standard to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 Follow these steps to create the data import file: Open the FileView program using the "FIVIEW" icon on the Windows Desktop. Select "Data" in left window. Select the data file of interest and double click to move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.30 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K2008.sbl" from this list of options and click "open." Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.31 Select "Quant Info" from the top menu and select "Quant Results" from the displayed options. This will display the data in concentration units.

7.32 Select the "Transpose" from the menu. Click on "file" within the chart to highlight the data.

7.33 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name to the file as "FileName.CSV", e.g., "JGA28A.CSV". Save the file to the ICP-MS DATA folder on metals on server_a.

7.34 Rename the pdf file to the appropriate file name in the PDF_Prints window and save to J-ICMS-Data file in My Network Places. Right click on ICPMS_CP.pdf icon to copy and paste blank file into PDF_Prints window for the next run.

To import data into the Metals Database:

7.35 Open the data file from metals on Server_a. Replace dashes in Cal Blank line with zeros. Replace dashes in Cal Std 1 line with 50 for most all elements. Change aluminum and silicon with 1000 and change sodium, magnesium, potassium, calcium, and iron with 10,000. All cells under metals with ###, replace with 999999. Save file in ICP-MS Data folder on Metals on Server a. Select the "ICPMS Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "JGA28A") and click "ok."

7.36 When the “Import finished” message appears, close the ICPMS Import window and select the “KIMS_METALS” icon from the Windows Desktop. The Metals Database Main Menu will appear. Select Additional Data Handling and then select Accept Samples by Element. Type in file name and reject any items that fail run QC.

7.37 Select the “Reporting Menu” button. From the Reporting Menu screen select the Batch QC Menu button and then the “Calculate Batch QC” button.

7.38 From the resulting list of QC results, deselect any items that fail run QC. Click on the “Accept Selected Batch QC” button.

7.39 From the Metals Main Menu, select the “Additional Data Handling” button. The Data Menu will appear. Select the “Report Added Compounds” button.

7.40 From the resulting list of sample results, deselect any items that fail run QC. Click on the “Accept Data” button.

7.41 Once all associated data from an analysis run have been processed, go to the RUNLOG INFO table of the metals database. Sort for the file of interest. Add lines for the 6020 and 200.8 Method Tunes. Change the time column accordingly. Go to the Generate Coverage portion of the Export Menu and print the Run Log and Logbook Page for the analysis run.

7.42 To extract Tune Reports and P/A Factor Tuning Report click on metpdf on Imageserver icon. Select J-ICPMS Data folder and select file on interest. Select Document drop down menu>pages>extract>select page numbers and click ok. Close document and save in metdpdf on “imageserver (P:) in J-ICP-MS-INST Tune folder as Filename+Tune.

7.43 Remove “blanks” and “rinses” from pdf file by selecting Document drop down menu>pages>delete>select appropriate pages at the beginning and end of report. Save document with ”RAW” added to the end of the file name. Save in the “ICPMS DATA” section of the “METPDF” directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific

judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least one multielement calibration standard. For each element,

calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples

analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as “estimated” with a “J”.

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high

concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

8.12 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12.1 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12.2 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.16 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine

the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

List of Tables

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	If more than 1 caibration std is used, correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit.
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike Sample (S), or Matrix Spike Duplicate Sample (P)	**At least one per digestion batch of 20 or fewer samples.**	**Recovery within ±25% of true value, if sample <4x spike added.**	**Flag results**
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section **8.13**	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 70%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	Daily.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration = 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICALhas passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low Level ICV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples,	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
			correct problem, and rerun all associated failed field samples.		
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. Flagging is not appropriate.	Results may not be reported without a valid calibration blank.	For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TABLE 2

DoD QSM 5.0/5.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).			
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-13	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 4

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	$^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571$ [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	$^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764$ [See note 3] $^{114}Cd = (114)*1 – (118)*0.0268 – (108)*1.84$ [See note 4]
	Calcium	Ca	44	$^{44}Ca = (44)*1 – (88)*0.0169$ [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	$^{54}Fe = (54)*1 – (52)*0.0282$ [See note 8] $^{57}Fe = (57)*1 – (43)*0.03$ [See note 9]
	Lead	Pb	206, 207, 208	$^{208}Pb = (208)*1 + (206)*1 + (207)*1$ [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	$^{98}Mo = (98)*1 – (99)*0.146$ [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	$^{82}Se = (82)*1 – (83)*1.009$ [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 – (53)*2.95 + (52)*0.378$ [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	$^{115}In = (115)*1 – (118)*0.016$ [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 4 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g “(51)”, indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W Solution (0.5mL each per 50mL and use same day only)	Inorganic Ventures	0.200
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Co, Cr, Mo, Mn, Pb, Sb, Sr, U, V	High Purity Standards	0.02
	10,000 mg/L Al, K, Na	High Purity Standards	0.002
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
	Conc. HNO_2	Baker Instra Analyzed	4
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (5% HNO_2/5%HCL)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Al, Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Th, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 -(5% HNO_2/5%HCL)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
	Conc. HCL	Baker Instra Analyzed	2
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each
ICP-MS-MIX-Y (1.0% HNO3/ 0.5% HCl)	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.030
	1000 mg/L As, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, V, Zn	High Purity Standards or Inorganic Ventures	0.30 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each
	1000mg/L Pb		0.30

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	CCV	Cal. Std.	ICV	PQL	P/A Tune Soln.
Aluminum	500.0	1000.0	400.0	20.0	200
Antimony	25.0	50.0	20.0	0.2	200
Arsenic	25.0	50.0	20.0	1.0	
Barium	25.0	50.0	20.0	0.4	
Beryllium	25.0	50.0	20.0	0.2	
Boron	25.0	50.0	20.0	4.0	
Cadmium	25.0	50.0	20.0	0.2	
Calcium	5000.0	10000.0	4000.0	20.0	
Chromium	25.0	50.0	20.0	1.0	200
Cobalt	25.0	50.0	20.0	0.2	200
Copper	25.0	50.0	20.0	0.6	
Iron	5000.0	10000.0	4000.0	20.0	
Lead	25.0	50.0	20.0	0.2	200
Magnesium	5000.0	10000.0	4000.0	20.0	
Manganese	25.0	50.0	20.0	0.4	200
Molybdenum	25.0	50.0	40.0	1.0	200
Nickel	25.0	50.0	20.0	0.4	
Potassium	5000.0	10000.0	4000.0	200.0	200
Selenium	25.0	50.0	20.0	1.0	
Silicon	500.0	1000.0	400.0	100.0	
Silver	25.0	50.0	20.0	0.2	
Sodium	5000.0	10000.0	4000.0	200.0	200
Strontium	25.0	50.0	20.0	1.0	200
Thallium	25.0	50.0	20.0	0.2	
Thorium	**25.0**	**50.0**	**20.0**	**0.2**	
Tin	25.0	50.0	20.0	1.0	
Tungsten	25.0	50.0	20.0	1.0	
Uranium	25.0	50.0	20.0	0.2	200
Vanadium	25.0	50.0	20.0	1.0	200
Zinc	25.0	50.0	20.0	2.0	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Thorium		**20**			
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Cerium					10.0
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	2.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	3.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Thorium		0.02			0.2	
Tungsten	5.0	0.10				0.2
Uranium		0.02			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TABLE 9 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 11

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2^{o} C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 12

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 13

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 14

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Yttrium (89)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Yttrium (89)
Calcium	44	Scandium (45)
Chromium	52	Yttrium (89)
Cobalt	59	Yttrium (89)
Copper	65	Yttrium (89)
Iron	57	Yttrium (89)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Yttrium (89)
Molybdenum	98	Yttrium (89)
Nickel	60	Yttrium (89)
Potassium	39	Scandium (45)
Selenium	82	Yttrium (89)
Silicon	29	Scandium (45)
Silver	107	Yttrium (89)
Sodium	23	Scandium (45)
Strontium	88	Yttrium (89)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Yttrium (89)
Zinc	66	Yttrium (89)

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in “Standard Methods for the Examination if Water and Wastewater,” Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-709
Revision History
Cover Page
Page 1

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tangney Date: 02/13/01

Operations Manager: C. Burton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2/13/01

General Manager: Date: 2/12/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 9045C	Format changes, added pollution prevention, database and operation of Accumet pH meter and calibration.	DN	2/13/01	2/13/01
04 9045C	Addition to Scope and Application to include reference for 9040B use when aqueous phase is >20%	DN	8/27/02	8/27/02
05 9045C	added KIMS Minor changes throughout added wording to sect. 6 New fig. 1 and 2	LAD	120104	120104
06 9045C	Added SW-846 reference. Minor formatting changes throughout.	LAD	03/07	03/07
07 9045D	Section 7.18 - Renamed "Equipment Maintenance" and revised for current practices. Add Wet Chem. Data Entry SOP reference. Updated references in section 10. Updated log book example.	LAD	08/09	08/09

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 9045D	Added references to sections 7 and 10.	LAD	06/10	06/10
09 9045D	Sect. 7.- Updated calibration procedure. Changed buffer pH probe stored in. Updated archived of reports. Added and edited references. Upgated Figures 1 & 2 added 3. Changed H.T. from ASAP to 28 days.	LAD	05/12	05/12
10 9045D	Sect. 6 - Updated holding time criteria. Sect. 7 - Updated calibration procedure to reflect current practice. Sect. 10 - Updated and added references. Updated Fig. 1 → 3	LAD	07/14	07/14
11 9045D	Sect. 7. Added requirement to reprep the sample if nostanding water is present.	LAD	08/16	08/16
12	Sect. 4 - added autotitrator, Sect. 5 - Removed old pH buffers for calibrations no longer used. Sect 7 - Added instructions for autotitrator operation, added export instructions for autotitrator. Added figures for soil prep logbook and autotitrator run. Updated references	LAD	10/19	10/19

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-709-12**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-709-12**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and techniques followed by Katahdin Analytical Services personnel to determine the pH of soils and waste samples in accordance with EPA method 9045 (current promulgated revision). Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample. If the aqueous phase is greater than 20%, pH determination should be performed in accordance with EPA method 9040 (current promulgated revision). Refer to the current revision of Katahdin SOP CA-708, pH Concentration Measurements in Aqueous Samples.

The procedures in this SOP are applicable to all non-CLP pH measurements performed for all soil matrices analyzed in the laboratory.

1.1 Definitions

pH - A measure of the acidity or alkalinity of a solution, defined as -log [H^+].

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pH in solids by EPA Method 9045. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the determination of pH concentration measurements in solid matrices to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for pH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a

potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention and Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A representative aliquot of sample, measured in grams, is mixed with an equivalent volume of laboratory reagent grade water, measured in mL. The solution is allowed to settle, and the pH of the standing water (decanted) is determined electrometrically.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated with an oily material. See section 7.18 for special cleaning instructions.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

4.0 APPARATUS AND MATERIALS

4.1 pH meter, Accumet Model 20 or equivalent with Automatic Temperature Compensation (ATC)

4.2 Glass beakers, 25 mL and 400 mL

4.3 25 mL dose cups

4.4 Teflon coated stir-bars

4.5 Stir-bar retriever

4.6 Magnetic stirplate

4.7 Shaker, 12 place

4.8 Analytical balance, capable of weighing to 0.1 g

4.9 Mettler Toledo Auto Titrator

4.10 Alkalinity Cups

4.11 125 mL or 250 mL plastic sample containers

5.0 REAGENTS AND STANDARDS

5.1 Buffer solutions (pH 2.0, 4.0, 7.0, 10.0, 12.0)

5.2 Laboratory reagent grade water (Lab Water)

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in soil jars and stored at 4˚C until analysis. Samples are collected in plastic or glass jars and stored at 4˚C until analysis.

pH samples require immediate analysis upon receipt by the laboratory.

SW846 Chapter 3 states the holding time for pH is “immediate”.

Katahdin project managers will remind clients that in order to meet the regulatory requirements for holding times, a field pH is required. If requested to perform a laboratory

pH, the analysis must be performed as soon as possible, and the data must be notated as being performed out of hold time.

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling. Mix samples thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Decant any standing liquid. Using the balance, weigh out 20.0 g of sample into a 125 or 250 mL plastic sample container. Record weight in soild prep logbook (Figure 1).

7.2 Add 20 mL of laboratory reagent grade water to the sample.

7.3 Place the sample on the shaker and allow it to shake, at medium speed, for five minutes. (CLP methods require the sample to shake for one hour.)

7.4 After five minutes (or one hour), remove the sample from the shaker and allow it to settle for one hour.

7.5 After one hour, decant the standing liquid into a 25 mL beaker. If no standing liquid is present, reprep the sample using 20g of sample and 40 mL of laboratory reagent grade water, cover with parafilm, and repeat steps 7.3 and 7.4. Alternatively sample can be placed on the centrifuge for 10 to 20 minutes.

7.6 Record total volume of laboratory reagent grade water added to sample in pH logbook. If volume of laboratory reagent grade water (in mL) added to sample exceeds the initial gram weight of the sample, flag sample data in pH logbook and record the reason for addition of excess laboratory reagent grade water (eg. minimum volume of water required in order to cover pH probe).

NORMAL RANGE CALIBRATION (pH range 3.5 – 10.5)

7.7 Meter should be calibrated daily. As described in the following steps, conduct a five-point calibration with pH buffers 2, 4, 7, 10 and 12. Perform a calibration check using pH 7 buffer. (In some cases, the entire five-point calibration is not needed in those cases run at least 3 of the calibration points). The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1). Mettler Auto Titrator is also calibrated daily with the pH4.0, 7.0 and 10.0 buffers. Before running the calibration on the Mettler Auto Titrator check the DI reservoir and top off in needed.

7.8 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.9 Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Then push *2,* to clear previous calibration. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.10 Remove pH 4 buffer. Rinse pH electrode and temperature probe. Blot dry.

7.11 Repeat step 7.1.3 with the pH 7 buffer. Record the value in the pH calibration logbook (figure 3). Remove pH 7 buffer. Rinse and dry pH electrode and temperature probe.

7.12 Repeat step 7.1.3 with the pH 10 buffer. Record the value in the pH calibration logbook (figure 3). Remove pH 10 buffer. Rinse and dry pH electrode and temperature probe.

7.13 To calibrate the Mettler Auto Titrator under series templates right click on pHcal and select run, then place 3 alkalinity cups with at least 20 mL of pH buffers 4, 7, 10 into slots 1-3. The slope of the calibration curve needs to be between -56 and -60.

NOTE: If any buffer readings are not within 0.05 pH units of expected values prior to calibration, the electrode may need cleaning. Note any maintenance performed and rerun the calibration.

LOW RANGE CALIBRATION

7.13 For samples with a pH less than 3.5, the meter must also be calibrated with pH buffer 2.

7.14 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.15 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.16 The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

HIGH RANGE CALIBRATION

7.17 For samples with a pH greater than 10.5, the instrument must also be calibrated using a ph buffer 12.

7.18 Rinse pH electrode and temperature probe with laboratory reagent grade water. Gently blot dry with kimwipe.

7.19 Place pH 12 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer. Push **Standardize** key. Push **Standardize** key, then push 1, to update. Enter value of pH buffer, once stabilized record the value in the pH calibration logbook (Figure 3).

7.20 The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

CALIBRATION CHECK / LABORATORY CONTROL SPIKE (LCS)

7.21 Place pH 7 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex, place pH electrode and temperature probe into buffer., but DO NOT press any keys as this reading is a calibration check. Record the reading in pH logbook as the LCS. Results must be within 0.05 pH units of the true value for analysis to proceed.

ANALYSIS OF SAMPLES

7.22 Sample analysis may proceed once the meter has been calibrated for the day with three buffers that bracket the expected pH of the sample.

7.23 Run the pH 7 buffer as the LCS for the analytical batch (Section 7.21). An LCS is required at the beginning of every batch of twenty or fewer samples.

7.24 Record date, time and initials for this analytical session.

7.25 The decanted samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers, □2 □C). A more accurate pH reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet™ pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet™ Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.

7.26 Pour about 25 ml of the supernatant into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.

7.27 When meter locks, record value displayed.

7.28 Rinse pH electrode and temperature probe. Blot dry

7.29 Place probe in pH 7 buffer solution to store until next analysis.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

ANALYSIS OF SAMPLES METTLER AUTO TITRATOR

7.30 After the Auto Titrator has been calibrated you may proceed with sample analysis.

7.31 Right click on PHTEST2 under the series template header and click run.

7.32 Change to number of samples to the appropriate number of samples and program in the samples with a CCV (20 mL pH7 buffer) at the beginning and every 10 samples and an LCS (20 mL pH 7 buffer separate source from CCV). (Note the instrument can only hold 18 cups per run).

7.33 Pour out each sample into an Alkalinity Cup making sure there is at least 20 mL of sample present (if not, reprep the sample with 20 g of sample and 40 mL of DI).

7.34 Click start.

EQUIPMENT MAINTENANCE

7.35 If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):

- be cleaned with an ultrasonic bath, or
- be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

An electrode that will not calibrate properly must be replaced.

REPORTING OF RESULTS

7.36 All pH measurements less than 10.0 are to be reported using two significant figures.

Examples: 2.46 = 2.5
6.32 = 6.3
9.94 = 9.9

7.32 All pH measurements which are at or greater than or round up to 10.0 are to be reported to three significant figures.

Examples: 9.95 = 10.0
12.25 = 12.3
13.76 = 13.8
11.95 = 12.0

7.33 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.

7.34 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.

7.35 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. Refer to the current revision of SOP CA-762 for instructions on data entry. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.36 All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

7.37 Once the Auto Titrator run is complete click the Results tab at the bottom of the page, click on the PH Test folder and find the run.

7.38 Right click on the run and hit Export Report save the report to the pH folder located in the Titrator Data folder.

7.39 Create a run log as shown in Fig 5 and print.

7.40 Work group samples and enter entry data from the soil prep logbook.

7.41 Remove CCV's and change the sample names of the LCS's and sample duplicates to the WG#'s assigned to them.

7.42 Save the file as a CSV (comma delimited) file then copy and paste that into the pH export folder then check and approve the results in KIMS.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client

and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 One sample duplicate is to be analyzed per batch or every 10 sample analyses.

8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results.

8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.

8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.

9.0 METHOD PERFORMANCE

Refer to method 9045.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, and IV, February 2007, Method 9045D.
Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS), current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March 2013

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Mettler Toledo Application M436-2006, Calibration of a pH Electrode.

Mettler Toledo Sample Changer, Terminal and Labx Operating Instructions.

LIST OF TABLES AND FIGURES

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 1

QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW9045	PH (soil)	3-5 point calibration with pH buffers with a midrange cal. check	Once per day, prior to use	± 0.1 pH units for each buffer Auto Titrator curve slope is between -56 to -60	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	± 0.1 pH units	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤ 20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-709-12	METHOD SW846 9045, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	1) Shake, at medium speed, for one hour. 2) Add more liquid after shaking and settling if there is no standing liquid left. 3) All buffers and samples are analyzed at room temperature. pH meter is equipped with automatic temperature compensation.	1) Continuously stir the suspension for five minutes. 2) No guidance for samples with no standing liquid left. 3) Report both pH and temperature at the time of analysis.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 1

EXAMPLE OF pH LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.

CORROSIVITY pH / pH Soil

Accumet 20 pH Meter - SN - C0024321 pH Probe SN - 20891000P

SW 846 9045D

CALIBRATION STDS:	CALIBRATED TO:	LOT NO:	NOTES:
pH 2.00	2.00	SWL 3617	WG147312
pH 4.00	4.00	3578	R284017
pH 7.00	7.00	3577	
pH 10.00	10.02	3579	
pH 12.00	11.98	3618	

LAB SAMPLE ID	ANALYSIS TIME	SAMPLE VOL (mL)	SAMPLE WEIGHT(g)	SAMPLE TEMP. (°C)	pH	REPORTED pH
LCS	15:11	—	—	22.3°C	7.00	7.0
SH5488-1	15:14	100	21.01	22.6°C	7.45	7.5
-1 Dup	15:18	"	21.91	21.9°C	7.67	7.8
SH5481-1	15:20	50	21.66	22.0°C	9.64	9.6
SH5494-1	15:22	*100	20.65	22.0°C	12.77	>12
SH5497-1	15:23	"	19.16	22.0°C	6.71	6.7
SH5585-1	15:26	50	20.85	22.0°C	6.73	6.7
-2	15:27	"	20.56	22.2°C	7.13	7.1
-3	15:31	"	20.37	22.0°C	7.24	7.2
-4	15:33	"	20.35	22.1°C	6.50	6.5
-5	15:35	"	19.97	22.2°C	6.69	6.7
CCV	15:37	—	—	22.5°C	7.02	7.0
SH5585-6	15:40	50	21.82	22.4°C	10.21	10.2
-6 Dup	15:46	"	20.98	22.6°C	9.71	9.7
-7	15:48	"	20.20	22.3°C	7.90	7.9
-8	15:51	"	20.05	22.3°C	7.44	7.4
-9	15:52	"	20.44	22.4°C	7.33	7.3
CCV	15:53	—	—	22.5°C	7.03	7.0
Blank	15:55	—	—	20.9°C	5.47	5.5
			ZS 7.29.14			

PREP ANALYST: ZS DATE/TIME: 7·28·14 16:00
ANALYST: ZS DATE: 7.29.14
CHECKED BY: DATE:

WL-015 - Revision 2 - 04/30/2012 QAWL693 0000099

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 2

EXAMPLE OF BATCH SHEET FOR pH

WET CHEMISTRY BATCH REPORT
Jul 15 2014, 01:21 pm
Batch: WG146539

Parameter: pH(Soil)
Prep Date: 15-JUL-14

Date Analyzed: 15-JUL-14
Prep Method: SW846 9045C

Analyst Initials: ZS
Prep Chemist: ZS

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SH4998-1	SAMP	SW846 9045D	10.83g	30mL	1	8.4	8.4 pH	82.	.1	0.10	0.10		
SH4998-2	SAMP	SW846 9045D	11.05g	30mL	1	7.58	7.6 pH	81.	.1	0.10	0.10		
SH5046-1	SAMP	SW846 9045D	10.51g	50mL	1	5.9	5.9 pH	22.	.1	0.10	0.10		
SH5074-3	SAMP	SW846 9045D	12.17g	30mL	1	9	9.0 pH	83.	.1	0.10	0.10		
SH5074-4	SAMP	SW846 9045D	11g	30mL	1	8.94	8.9 pH	81.	.1	0.10	0.10		
SH5150-1	SAMP	SW846 9045D	23.64g	100mL	1	7.03	7.0 pH	14.	.1	0.10	0.10		
WG146539-1	LCS	SW846 9045D	20g	20mL	1	7	7.0 pH	NA	.1	0.10	0.10		100
WG146539-2	DUP	SW846 9045D	10.99g	30mL	1	9.08	9.1 pH	NA	.1	0.10	0.10	2	
WG146539-3	MBLANK	SW846 9045D	20g	20mL	1	5.69	5.7 pH	NA	.1	0.10	0.10		

Comments:

SH5074-3	NTC-NYCDEP-SED-SPLIT-21; MS/DUP on metals and cyanide. DUP on Black Carbon, TS, pH. MS/MSD on Ammonia, TKN, N+N and Total P. Can be moved to another sample if needed.
SH5074-4	NTC-NYCDEP-SED-SPLIT-22; MS/MSD on DORO and SVOC;MS/DUP on TOC. Can be moved to another sample if needed.
WG146539-1	SH5074-4
WG146539-2	SH5074-4
WG146539-3	SH5074-4

Entered by: ZS Date: 7.15.14 Accepted by: [signature] Date: 07/15/14

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 3

EXAMPLE OF PH CALIBRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

pH METER CALIBRATION RECORD

FISHER ACCUMET 20 - SERIAL NUMBER C0024321

DATE	INITIALS	pH TRUE VALUES AND ACCEPTABILITY														
		2.00	± 0.05 (3)	Lot #	4.00	± 0.05 (3)	Lot #	7.00	± 0.05 (3)	Lot #	10.00	± 0.05 (3)	Lot #	12.00	± 0.05 (3)	Lot #
6-16-14	BN	2.00	✓	SWL 3617	3.96	✓	SWL 3578	7.00	✓	SWL 3577	10.00	✓	SWL 3579	11.95	✓	SWL 3618
6-17-14	BN	1.98	✓	"	3.96	✓	"	7.01	✓	"	9.99	✓	"	11.95	✓	"
6-18-14	ZS	2.00	✓	"	4.00	✓	"	7.00	✓	"	10.00	✓	"	12.03	✓	"
6/19/14	UNP				3.99	✓	"	6.98	✓	"	9.99	✓	"	12.00	✓	"
6/20/14	UNP				4.00	✓	"	6.98	✓	"	9.98	✓	"	12.03	✓	"
6/23/14	UNP	1.99	✓	"	3.98	✓	"	6.99	✓	"	9.99	✓	"	12.01	✓	"
06-24-14	BN	2.00	✓	SWL 3617	4.00	✓	"	7.03	✓	"	10.03	✓	"	11.97	✓	"
6-25-14	DW			↓	4.00	✓	"	7.00	✓	"	10.00	✓	"			
06/26/14	[illegible]	2.01	✓	"	4.00	✓	"	7.00	✓	"	9.99	✓	"			
6.27.14	ZS	2.00	✓	"	3.96	✓	"	6.99	✓	"	9.99	✓	"	12.00	✓	SWL 3618
6/30/14	UNP	2.00	✓	"	3.97	✓	"	6.99	✓	"	9.99	✓	"	12.01	✓	"

MAINTENANCE – Include date, initials and task

QA-070 – Revision 1 – 09/30/2010

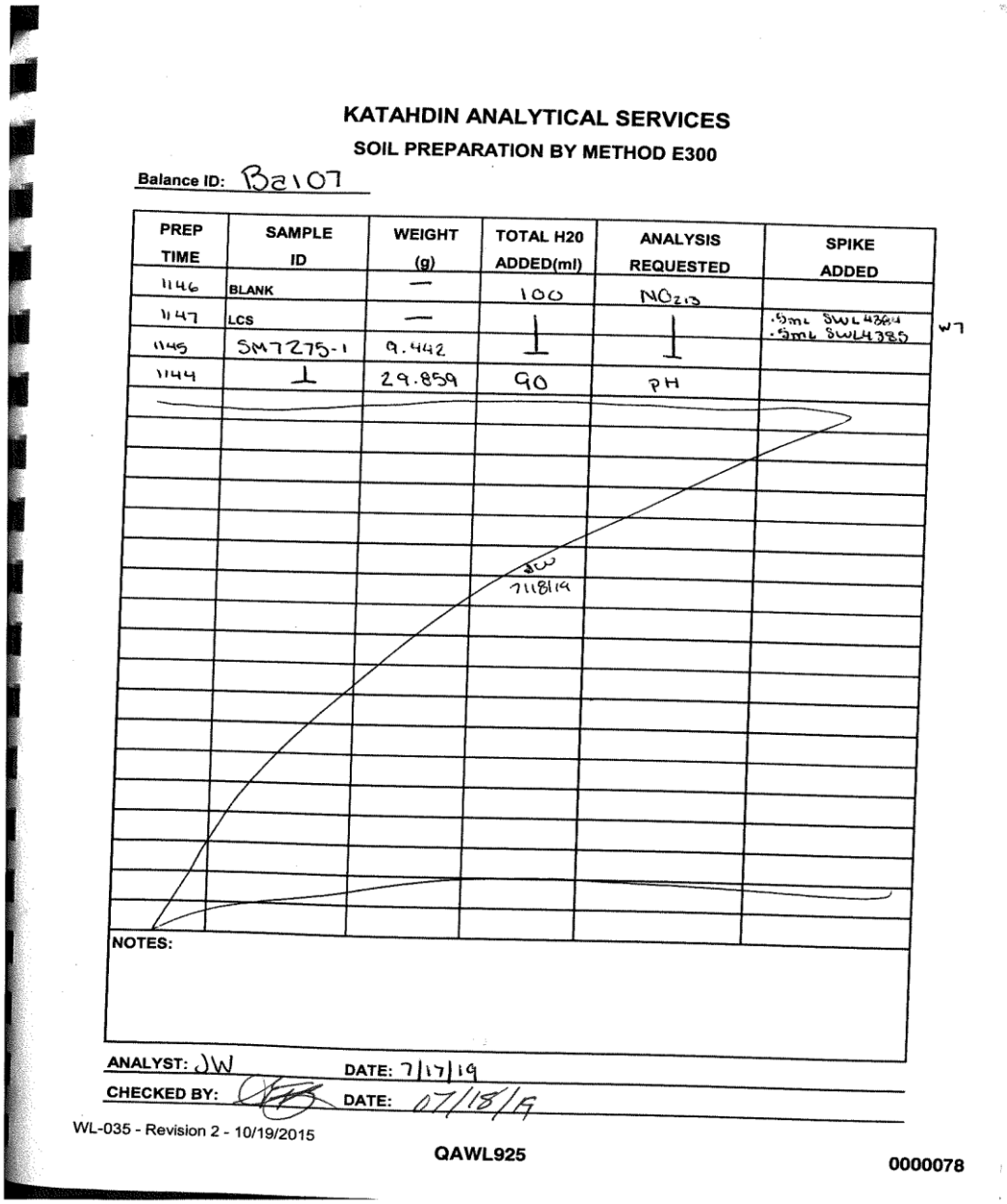
QAQC615

0000039

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 4

EXAMPLE OF SOIL PREP LOGBOOK



KATAHDIN ANALYTICAL SERVICES

SOIL PREPARATION BY METHOD E300

Balance ID: B2107

PREP TIME	SAMPLE ID	WEIGHT (g)	TOTAL H20 ADDED(ml)	ANALYSIS REQUESTED	SPIKE ADDED
1146	BLANK	—	100	$NO_{2:3}$	
1147	LCS	—	⊥	⊥	.5mL SWL4386 .5mL SWL4385 W7
1145	SM7275-1	9.442	⊥	⊥	
1144	⊥	29.859	90	pH	

JW 7/18/19

NOTES:

ANALYST: JW DATE: 7/17/19

CHECKED BY: DATE: 07/18/19

WL-035 - Revision 2 - 10/19/2015

QAWL925

0000078

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 5

EXAMPLE OF AUTO TITRATOR RUN

E150.1 WG757209 RS14059
SM4500HB WG757210 RS14060
SW9045C WG757212 RS14061

Start time: 7/17/2019 16:09
Results

No.	Comment / ID	Start time	Rx	Result	Unit	Name	%RECVY
1/12	CCV	7/17/2019 4:10:45 PM	R3 =	7.029	pH	pH	100.41%
			R4 =	23.3	oC	TEMPERATURE	
2/12	LCS	7/17/2019 4:15:17 PM	R3 =	7.048	pH	pH	
			R4 =	22.5	oC	TEMPERATURE	
3/12	SM7275-1	7/17/2019 4:19:43 PM	R3 =	7.551	pH	pH	
			R4 =	22.7	oC	TEMPERATURE	
4/12	SM7275-2	7/17/2019 4:24:10 PM	R3 =	7.772	pH	pH	
			R4 =	22.2	oC	TEMPERATURE	
5/12	SM7253-1	7/17/2019 4:28:37 PM	R3 =	7.124	pH	pH	
			R4 =	21	oC	TEMPERATURE	
6/12	SM7303-1	7/17/2019 4:33:04 PM	R3 =	7.916	pH	pH	
			R4 =	19.3	oC	TEMPERATURE	
7/12	SM7303-1 DUP	7/17/2019 4:37:32 PM	R3 =	7.9	pH	pH	
			R4 =	19.3	oC	TEMPERATURE	
8/12	SM7303-2	7/17/2019 4:41:59 PM	R3 =	8.066	pH	pH	
			R4 =	19.4	oC	TEMPERATURE	
9/12	SM7303-3	7/17/2019 4:46:27 PM	R3 =	7.892	pH	pH	
			R4 =	19.5	oC	TEMPERATURE	
10/12	SM7303-4	7/17/2019 4:50:54 PM	R3 =	7.713	pH	pH	
			R4 =	20.1	oC	TEMPERATURE	
11/12	SM7303-5	7/17/2019 4:55:20 PM	R3 =	8.296	pH	pH	
			R4 =	20.5	oC	TEMPERATURE	
12/12	CCV	7/17/2019 4:59:47 PM	R3 =	7.031	pH	pH	100.44%
			R4 =	22.2	oC	TEMPERATURE	

Start time: 7/17/2019 14:57
Results

No.	Comment / ID	Start time	Rx	Result	Unit	Name
Calibration data						
	Number of segments		1	pH 4:	SWL	4477
	Slope	-57.80 mV/pH		pH 7:	SWL	4489
	Zero point	7.064 pH		pH 10:	SWL	4458
	Calibration temperature	23.7 oC		LCS:	SWL	4476

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-733
Revision History
Cover Page
Page 1

TITLE: **REACTIVE CYANIDE: SW-846, 7.3.3.2**

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tanguay Date: 02.13.01

Operations Manager: Jol C. Burton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2.13.01

General Manager: Dennis F. Mahan Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, database and new CN SOP reference. Other minor changes throughout.	DN	2.13.01	2.13.01
02	major changes to apparatus and procedure to reflect current practice. Added reference for SOP CA-107 to sect. 1.4. Added program specific info to Sect. 8. Supervisor ⇒ dept. manager, ASTM II water ⇒ lab. reagent grade H_2O. Updated figures	LAD	04/06	04/06
03	Section 7.1.3 and 7.1.4 Changed spike amount from 0.1 mL to 0.05 mL. Added definitions to section 1.	LAD	02/08	02/08
04	Sec. 1.4 - Changed "G" Stream to "N-High" stream. Sect. 20 - Changed SOP reference. Removed Secs 4.7 and 4.10. Sect. 4.13 - changed to Eppendorf. Sect. 5.3 - Added purchased with certified reference value option. Added DOC and MDL criteria to Table 1. Added 2 method deviations to Table 2.	LAD	05/09	05/09
05	Added definitions to section 1.1. Revised Table 1. Added EHSM, Subsampling, QA-86, DoD, NELAC and CA-101 references.	DN	08/09	08/09

TITLE: **REACTIVE CYANIDE: SW-846, 7.3.3.2**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Minor changes to sections 5, 7 and 8 to reflect current standard volumes concentrations and prep. Added CCB and CCV to Table 1 Added References	LAD	07/11	07/11
07	Sect. 7. updated LCS and MS spike amounts Sect. 8- Added contingency plan. Sect. 10- updated References Updated figures 1 and 2.	LAD	05/18	05/18

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-733-07, titled REACTIVE CYANIDE: SW-846, 7.3.3.2.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-733-07, titled REACTIVE CYANIDE: SW-846, 7.3.3.2.

Recipient: _____ Date: _____

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

1.0 SCOPE AND APPLICATION

The intended application of this method is to determine the hydrogen cyanide released from wastes. This method is applicable to all waste except those that will form explosive mixtures when combined with acids. This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolved under the test conditions. The regulatory limit for *Total Releasable Cyanide* is 250 mg/Kg waste.

1.1 Definitions

Reactive Cyanide - Cyanide released under the test conditions defined under SW846 Chapter 7, 7.3.3.2 where the sample is exposed to mildly acidic conditions.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

LCS - Laboratory Control Sample - A standard or solid reference material of known value that has been brought through the sample preparation an analysis process. The LCS is used to assess the accuracy of the method.

LOD – Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

MB – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of reactive cyanide according to SW-846, 7.3.3.2. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability”, current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of reactive cyanide according to SW-846, 7.3.3.2, to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.
It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure. These materials include the following: Sodium Hydroxide, Potassium Cyanide, Sulfuric Acid, Hydrochloric Acid, Barbituric Acid, Silver Nitrate and Pyridine.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health & Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

All remaining basic waste from the distillation, receiver contents, is treated as though cyanide is present and disposed of in the pyridine (“N-High” stream) waste satellite located in the Wet Chemistry laboratory. When this container is full, it is then taken to the hazardous waste disposal area and the contents are transferred to the pyridine waste drum.

The acidic portion of the distillation, still contents, is placed in acid waste (“A” stream) via the satellite accumulation in the Wet Chemistry laboratory. Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety

Manual and SOP SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards,” current revisions. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

An aliquot of acid is added to a fixed weight of waste in a closed system. The gas that is generated is swept into a scrubber. The cyanide in the gas is absorbed in a NaOH scrubbing solution that is analyzed for cyanide by Katahdin SOP CA-773, “Colorimetric Analysis of Total and Amenable Cyanide Using the Automated Konelab Multiwavelength Photometric Analyzer”.

3.0 INTERFERENCES

Interferences are undetermined.

4.0 APPARATUS AND MATERIALS

4.1 Flexible tubing for connection from the nitrogen supply to the apparatus and from the flask to the absorber impinger unit (scrubber).

4.2 Nitrogen gas tank with regulator.

4.3 Gas valve capable of metering N_2 flow to 20 psi

4.4 Flowmeter capable of measuring flow at 60 mL/min at the distillation station.

4.5 Analytical balance weighing to 0.001g.

4.6 10-mL buret

4.7 12 gas washing bottles with 250ml graduated cylinders

4.8 Buret stand and holder

4.9 0.1mL Eppendorf pipet and tips

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5.0 REAGENTS AND STANDARDS

5.1 Sulfuric Acid (0.1 N), H_2SO_4: Add 5.6 ml of concentrated H_2SO_4 to laboratory reagent grade water and dilute to 2 liters.

5.2 Sulfuric Acid (0.01 N), H_2SO_4: Volumetrically transfer 200 ml of 0.1 N H_2SO_4 and dilute to 2 liters with laboratory reagent grade water to make the 0.01 N H_2SO_4.

5.3 Stock Cyanide Solution (1000 mg/L): purchase as certified solution

5.4 Sodium Hydroxide Solution (0.25 N) ,NaOH: Dilute 25.0 ml of 10 N NaOH to 1 liter of Laboratory reagent grade water. This solution could also be made by dissolving 10 g of NaOH in Laboratory reagent grade water and diluting to 1 liter.

5.5 Laboratory reagent grade water: Equivalent in protocol as reagent or DI water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Samples should be collected with a minimum of aeration. The filled sample bottle should contain no headspace and should be kept cool and in the dark until analysis. Samples can be held 14 days with no preservative. Perform analysis in a ventilated hood.

6.2 Samples can be preserved by adjusting the sample pH to 12 with strong base; however, this will cause dilution of the sample, increase the ionic strength, and possibly change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid.

7.0 PROCEDURES

7.1 PREPARATION OF SAMPLE

7.1.1 Weigh approximately 10 g of sample in a 250 mL addition graduated cylinder. Record weight in the preparation logbook (Figure 1).

Note: Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.1.2 To prepare Method Blank transfer 10 g of laboratory reagent grade water to a 250 mL addition graduated cylinder

7.1.3 To prepare LCS transfer 0.2 mL (50 μg CN) of the Stock Cyanide Solution (1000 mg/L, See Reagents 5.3) to a 250 mL addition graduated cylinder.
Note: Standard should be added last. Be sure to seal the gas washing bottle quickly after addition.

7.1.4 To prepare a Matrix Spike (MS), weigh approximately 10 g of sample in a 250 mL addition graduated cylinder. Spike the sample with 0.2 mL (50 μg CN) of the stock cyanide solution (1000 mg/L, see reagent 5.3). (See LCS note)

7.1.5 To prepare sample Duplicate weigh out approximately 10 g of the sample selected/designated as the sample duplicate in a 250 addition graduated cylinder

7.1.6 Add 190 ml of 0.25 N NaOH to each of the absorber graduated cylinders. Place all scrubbers in graduated cylinders. Connect nitrogen hoses to scrubbers.

7.1.7 Add 180 mL of 0.01 N H_2SO_4 to each addition graduated cylinder. Immediately seal all graduated cylinders.

7.1.8 Turn on the main valve on the Nitrogen tank. Make sure it is reading 300 psi or greater.

7.1.9 Adjust the local N_2 pressure valve in the hood ting knob and set the pressure to 20 psi on the low pressure gauge.

7.1.10 Turn the Outlet Valve on from the flowmeter on until the flow registers 60 mL/min.

7.1.11 Use timer set for 30 minutes. After 30 minutes, close off the main valve on the nitrogen tank followed by the pressure adjusting knob and then the outlet valve. Disconnect all of the scrubbers on the apparatus.

7.1.12 A portion of the scrubber is transferred to 40-mL VOA vial for CN analysis. The remainder (150 mL) is covered and titrated ASAP for reactive sulfide where requested.

7.2 ANALYSIS OF CN

7.2.1 Cyanide concentration in the scrubber is determined by automated colorimetry (e.g., Konelab) in accordance with the protocols delineated in the most current revision of Katahdin SOP CA-773, Total Cyanide, for the analysis procedure.

7.2.2 A portion of the scrubber solution may also be used for Reactive Sulfide analysis. See SOP CA-734, Reactive Sulfide: SW-846, 7.3.4.2.

7.2.3 The rate of release of HCN (mg/Kg/sec) is calculated as follows:

$$R = \text{Specific Rate or Release, mg/Kg/Sec} = \frac{A \times V}{W \times S}$$

where: A = concentration of HCN in the scrubber as mg/L
= 1.04 x CN mg/L (1.04=MW HCN/MW CN= 27.03/26.0179)
V = volume in scrubber, Liters, i.e. 0.19
W= weight of waste, Kg
S = Time of measurement, Time N_2 stopped - Time N_2 started, sec

7.2.4 The releasable HCN as mg/Kg is calculated as follows:

$$\text{Total Releasable HCN, mg/Kg} = \frac{A \times V}{W}$$

where: A = concentration of HCN in the scrubber as mg/L
= 1.04 x CN mg/L (1.04=MW HCN/MW CN= 27.03/26.0179)
V = volume in scrubber, Liters, i.e. 0.19
W= weight of waste, Kg

7.3 REPORTING

7.3.1 Enter results, including sample preparation information, measured sample concentrations, and quality control data, into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.3.2 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents

and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments.

These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Every instance of noncompliant method quality control requires the generation of a Corrective Action Report describing the problem, suspected cause and final resolution. Corrective action reports must be signed by the initiator, Department Manager, QA officer, and lab management.

8.1 One laboratory control sample (LCS) is distilled with every batch of 20 samples. The LCS spike solution (1000 mg/L cyanide standard) is an independently prepared standard from which 0.05 ml is distilled. Evaluate the % recovery based on historical laboratory data. The range of recovery for the LCS is 0 – 100 %.

8.2 A duplicate is run every ten samples. Sample duplicates are expected to agree within 20% relative difference. If duplicate samples are out of control, re-distill another replicate.

8.3 A method blank is analyzed with every batch or analytical session. The concentration of the blank must be less than the detection limit (1 mg/kg).

8.4 Non-conformance report: Every instance of noncompliant method quality control requires the generation of a non-conformance report describing the problem, suspected cause and final resolution. Non-conformance reports must be signed by the initiator, Inorganic Department Manager, QA officer, and lab management.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

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9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined initially prior to sample analysis and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of SW-846, 7.3.3.2 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Chapter Seven, Section 7.3.3.2, Rev. 4, September 2004.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 9012, Rev. 0, September 1986.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

Katahdin SOP CA-773, “Colorimetric Analysis of Total and Amenable Cyanide Using the Automated Konelab Multiwavelength Photometric Analyzer".

Katahdin SOP CA-101, “Equipment Maintenance and Troubleshooting”, current revision.

Katahdin SOP CA-762, “Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)”

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

List of Figures and Tables

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Reactive Cyanide SW-846 7.3.3.2	Method blank	One per prep batch of 20 or fewer samples	HCN not detected >PQL (For DoD QSM, no analyte detected > ½ PQL and > 1/10 the amount measured in any sample)	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.
	CCV	At beginning of run, after every ten samples, and at end of run	85-115% recovery	(1) If the CCV fails high, report samples that are <PQL (2) Recalibrate and/or reanalyze other samples
	CCB	Immediately following each CCV	No analyte detected >PQL	(1) Investigate source of contamination (2) Report all sample results >10x the CCB (3) Report all sample results <PQL (4) Reanalyze all other samples associated with failing CCB
	LCS	One per prep batch of 20 or fewer samples	0 – 100% nominal; statistically derived after sufficient historical	(1) If the LCS fails high, report samples that are <PQL. (2)Reanalyze /or recalibrate and reanalyze (3) Redistill, recalibrate and/or reanalyze other samples.
	Matrix Spike	One per prep batch of 20 or fewer samples	0-100 %R	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Analyze unspiked sample scrubber solution with post-scrub spike to confirm matrix interference present in the scrubber (4) It should be anticipated that 0% to very low recoveries may be evidenced in high metals content samples. (5) Notate sample result in raw data if matrix interference confirmed
	Sample Duplicate	One every ten samples	RPD $\leq$ 20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.
	Demonstration of analyst proficiency	One time per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-733-07	Method 7.3.3.2, current revision
Apparatus/Materials	(1) 250 mL scrubber (2) Gas washing bottles	(1) 50 mL scrubber (2) Ground glass glassware
Reagents	(1) Cyanide reference solution, 1000 mg/L prepared in 250 mL with 0.5 g KOH	(1) Cyanide reference solution, 1000 mg/L prepared in 250 mL with 0.625 g KOH
Sample preservation/ handling		
Procedures	(1) 190 mL of 0.25 N NaOH are added to each scrubber (absorber bottle) prior to distillation. (2) 180 mL of 0.01 H2SO4 added to reflux bottle.	(1) 50 mL of 0.25 N NaOH is added to each scrubber, then diluted with water to obtain an adequate depth of liquid in the scrubber. (2) Add enough sulfuric acid to fill flask half full.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF LOGBOOK PAGE

KATAHDIN ANALYTICAL
REACTIVE SULFIDE W6722604B9

METHOD: SW846 9030 / 7.3.4.2				PQL: 27 mg/kg				
REAGENT LOT #:								
IODINE STANDARD: WI6109				STS SOLUTION-0.0375N: SWL4057				
Na2S SOLUTION: WI6300				NaOH-0.25N: WI6319				
HCl-6N: WI6336 / WI6259				H2SO4 ~~.005N~~: 0.01N / WI6320				
Total CN (1000ug/ml) SWL4074				Starch indicator soln: WI6345				
LCS PREP:	10mL WI6300 (WI3) + 0.2mL SWL4274 WI8							
MS PREP:	↓	↓	↓	↓	↓			

STANDARDIZATION OF I2		CALC OF I2 N	STANDARDIZATION OF Na2S			CALC OF Na2S mg/ml
VOL(ml) I2	VOL(ml) Na2S2O3		VOL(ml) I2	VOL(ml) Na2S2O3	VOL(ml) Na2S	
10	6.35		10	2.90	2	
10	6.35		10	3.00	2	
10	6.30		10	2.85	2	
	X:			X:		

Prep Start Time	Prep End Time	Sample ID	Sample Wt. (g)	NaOH Trap Vol.(ml)	Analysis Vol.(ml)	ml I2 Soln Added	ml STS to Endpoint	Time of Analysis
1020	1050	Blank	—	190	150	10	6.35	1333
		LCS	—			25	5.20	1337
		93081-1	11.176			10	6.40	1342
*		93089-1	11.087			10	6.40	1345
		DUP	11.276			10	6.40	1350
↓	↓	MS	11.150	↓	↓	25	5.60	1341
1513	1543	93118-1	11.460	190	150	10	6.10	1635
*	↓	↓ 2	11.681	↓	↓	10	6.10	↓ 1640

4/17/18

NOTES: * WI3

ANALYST: SC DATE: 4.12.18
CHECKED BY: DATE: 04/18/18

WL-014 - Revision 2 - 03/08/2017 QAWL848 0000078

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FIGURE 2

EXAMPLE OF BATCH SHEET

WET CHEMISTRY BATCH REPORT
Apr 17 2018, 03:58 pm
Batch: WG226497

Parameter: Cyanide, Reactive
Date Analyzed: 13-APR-18
Analyst Initials: ZF

Prep Date: 13-APR-18
Prep Method: SW846 7.3.4
Prep Chemist: SC

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SL3034-1	SAMP	SW846 7.3.3	10.87sg	190.00mL	.954	.14571	U10. mg/Kg	NA	20	0.16	20.		
SL3069-1	SAMP	SW846 7.3.3	11.037g	190.00mL	.906	.43077	U10. mg/Kg	NA	20	0.16	20.		
SL3118-1	SAMP	SW846 7.3.3	11.460g	190.00mL	.977	.71517	U10. mg/Kg	NA	20	0.16	20.		
SL3118-2	SAMP	SW846 7.3.3	11.431g	190.00mL	.88	.17738	U10. mg/Kg	NA	20	0.16	20.		
WG226497-1	MBLANK	SW846 7.3.3	10.000g	190.00mL	1	+.91812	U10. mg/Kg	NA	20	0.16	20.		
WG226497-2	LCS	SW846 7.3.3	10.000g	190.00mL	1	666.6699	J13. mg/Kg	NA	20	0.78	100		68
WG226497-5	DUP	SW846 7.3.3	11.276g	190.00mL	.887	.2686	U10. mg/Kg	NA	20	0.16	20.	NC	
WG226497-6	MS	SW846 7.3.3	11.190g	190.00mL	4.464	483.056	J8.4 mg/Kg	NA	20	0.78	90.		43

Comments:

WG226497-1	SL3069-1
WG226497-2	SL3069-1
WG226497-3	SL3069-1
WG226497-4	SL3069-1

Entered by: ZF Date: 4/17/18 Accepted by: [signature] Date: 04/18/18

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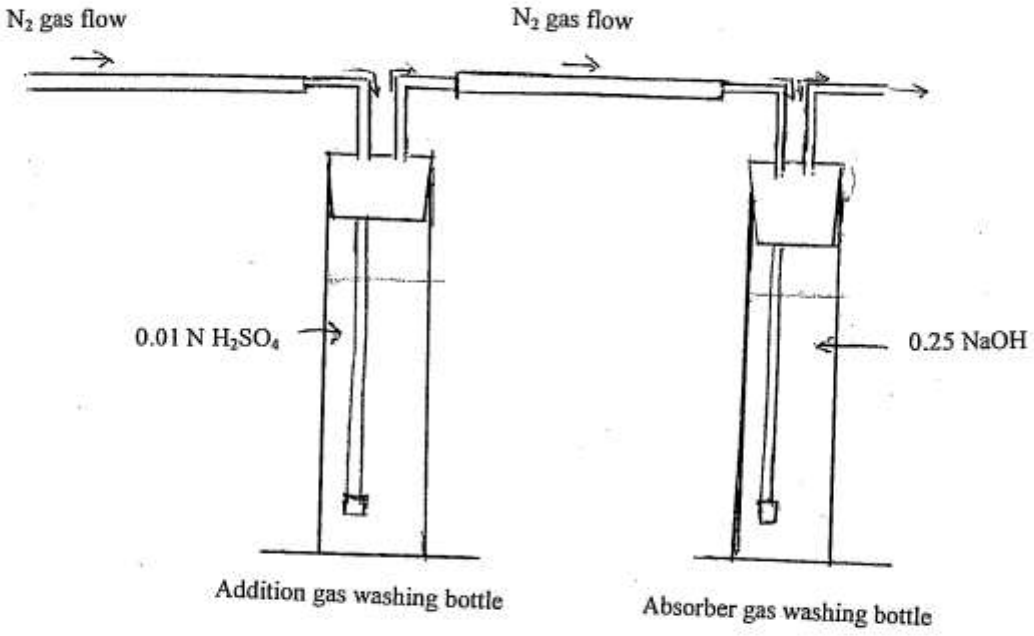
FIGURE 3

REACTIVE CYANIDE APPARATUS



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Alex Pimentel

Review Date: 1/10/19

SOP Number: CA – 733 – 07

SOP Title: REACTIVE CYANIDE: SW-846, 7.3.3.2

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: T. Brewer **Date:** 01/24/19

QAO Signature: Jessie Dimond **Date:** 01.24.19

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-736
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TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

Prepared By: Betsy Carbone Date: 8/96

Approved By:

Group Supervisor: Keith Longley Date: 01/24/01

Operations Manager: [signature] Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: [signature] Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, p-xylene in duplicate, correction for barometric pressure	DN	1.22.01	1.22.01
02	Many changes throughout to reflect current practices. New fig. 2	LAD	031805	031805
03	Added method blank and LCS definitions	LAD	06/08	06/08
04	Added definitions.	LAD	09/10	09/10
05	Sect. 7 - Change increase temperature to 5±6°C/min to set instrument to correct matrix. Also, added raw data archival info. Sect. 10 - Added and edited references. Updated Figures 1 and 2.	LAD	05/12	05/12

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 2 and 7 – Added Method B for Suspensions of solids and highly viscous materials. Sect. 1 and 10 – Updated method references. Sect. 7 – Added using organic solvents may be used to clean cup. Changed KAS INC to KAS	LAO	08/15	08/15
07	Changed temperature interval from 2°C to 1°C. Removed method modification from Table 2. Specified soil results should be reported as Method 1010 modified. Updated references	LAO	07/19	07/19

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-736-07**, titled **TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-736-07**, titled **TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER**.

Recipient: _____ Date: _____

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services laboratory personnel to measure the tendency of a sample to form a flammable mixture with air under controlled laboratory conditions. The objective of the ignitability characteristic is to identify wastes that either present fire hazards under routine storage, disposal, or transportation or are capable of severely exacerbating a fire once started. The SOP is applicable to SW-846 method 1010A and ASTM method D 93-79.

The test method covers the determination of the flash point by Pensky-Martens closed-cup tester for fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions and other liquids.

1.1 Definitions

Flash Point - The lowest temperature of the sample, corrected to a barometric pressure of 760 mm of Hg, at which application of the test flame ignites the vapor above the sample.

Laboratory Control Sample (LCS): LCS is a known standard carried through the entire analytical procedure in the same manner as a sample. The LCS determines the validity of the batch.

Method Blank - A Laboratory Reagent Grade Water sample that is carried through the entire analytical procedure in the same manner as a sample.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in flashpoint analysis by Pensky-Martens Closed-Cup method. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in flashpoint analysis by Pensky-Martens Closed-Cup method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

p-Xylene must be stored separately and disposed of as a flammable liquid. All sample residues under this protocol are disposed of in satellite wastes for flammable liquids. Other wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

2.0 SUMMARY OF METHOD

Samples are heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample. For most samples, Method A is used to determine flash point, for suspensions of solids and highly viscous materials, Method B should be used.

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

3.0 INTERFERENCES

None determined.

4.0 APPARATUS AND MATERIALS

4.1 Pensky-Martens Closed-Cup Flash Tester

4.2 Calibrated thermometer capable of reading up to 120°C

4.3 Barometer

5.0 REAGENTS

5.1 p-Xylene Reference Standard - Reagent grade, Flash point 27°C

5.2 Laboratory Reagent Grade Water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Record the ambient barometric pressure in inches of mercury. Multiply this number by 25.4 mm/in to obtain the barometric pressure in mm of mercury. Record this number in the appropriate place in the logbook (figure 1).

7.2 Preparation of Apparatus - Place the tester on the bench top located under a fume hood. Although the hood is turned off while performing the analysis, a draft is still present. The tester must be surrounded on three sides with a shield that is sufficient enough to prevent sputtering of the pilot flame.

7.3 Preparation of Sample - Samples of very viscous materials must be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. Samples shall never be heated above a temperature of 17°C below the expected flash point.

7.4 Analytical Procedure – Method A, Basic Procedure

7.4.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus. Organic solvents (methylene chloride, hexane) may be used in clean cup. Additional cleaning may be accomplished with the aid of sand or sandpaper.

7.4.2 Check to be sure that the orifice for the flame wick is not clogged. A piece of wire should fit into the opening.

7.4.3 For aqueous samples, fill the cup with the sample to be tested to the level indicated by the filling mark. For solid samples, fill the cup with the sample to be tested to the level indicated by the filling mark. Results for solids samples should be reported under Method 1010 (Modified). Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for information on subsampling.

7.4.4 Place the lid on the cup and set the cup in the apparatus stove. Be sure to have the locking or locating device properly engaged.

7.4.5 Insert the thermometer.

7.4.6 If the flashpoint is known to be high, bring the material to be tested and the tester to a temperature of 25 $\pm$ 5°C or 15 $\pm$ 5°C lower than the estimated flash point, whichever is lower. Otherwise, start the flame when the samples are still cold (below room temperature)

7.4.7 Light the test flame and adjust it to 5/32 inch (4mm) in diameter.

7.4.8 Turn the heating dial to the black mark which corresponds to the sample matrix being tested.

7.4.9 Turn the stirrer on (90-120 rpm), stirring in a downward direction.

7.4.10 Apply the test flame when the temperature is between 20 and 25°C.

7.4.11 Apply the test flame by operating the mechanism on the cover that controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 seconds, left in its lowered position for 1 second and quickly raised to its high position.

NOTE: Do not stir the sample while applying the test flame.

7.4.12 After 25°C, apply the test flame in increments of 1°C.

7.4.13 Continue applying the test flame at temperature increments of 1°C until the flash point of the sample or 71°C is reached, whichever comes first.

7.4.14 If the sample flashes between 25 and 71°C, obtain a fresh aliquot of the sample. Bring the sample material to a temperature 15 ± 5°C lower than the initially determined flash point. Apply the test flame and thereafter at temperature readings in increments of 1°C until the flash point of the sample is reached. Results obtained from Steps 7.4.13 and 7.4.14 should agree within ±2 °C.

7.4.15 Record the observed flash point as the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. The lowest reading from the duplicate analyses (Steps 7.4.13 and 7.4.14) should be reported.

NOTE: Do not confuse the true flash with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

7.4.16 If the sample flashes below 25°C, the reported value should be <25°C. If the sample did not flash, the reported value should be >71°C. If the sample was not heated to 71°C, record the highest temperature achieved.

7.4.17 The observed flash points must be corrected for the ambient barometric pressure. If the ambient barometric pressure at the time of analysis differs from 760 mm Hg (one atmosphere), the following formula must be used:

Corrected flash = (observed flash + 0.033 (760 mm Hg – ambient barometric
Point (° C) point in ° C) pressure in mm Hg)

Record all corrections in the logbook (Figure 1).

7.4.18 After completion of each test, the logbook must be signed and dated by the person performing the test.

7.4.19 The sample data results from the logbook, with any appropriate notations, are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. Refer to the current revision of Katahdin Analytical Services SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.5 Analytical Procedure – Method B, Determination of Flash Point of Suspensions of Solids and Highly Viscous Materials.

7.5.1 Follow steps in section 7.4 **except -**

7.5.1.1 Bring the sample material to a temperature 15 ± 5°C or 11°C lower than the estimated flash point, whichever is lower.

7.5.1.2 Increase the stirrer speed to 250 +/- 10 rpm

7.6 Archival of Raw Data

All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A typical analytical run consists of a tester calibration using p-Xylene (analyzed in duplicate), a blank consisting of laboratory reagent grade water (immediately following the p-Xylene), the samples to be analyzed and a duplicate sample analysis. A duplicate sample analysis is performed every ten samples, every daily batch, or for any sample that flashes, whichever is more frequent. If a sample flashes, that sample is run in duplicate. Refer to Table 1 for acceptance criteria and corrective actions.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

Refer to the current revision of Method 1010 for method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM, Test Methods for Flash Point by Pensky-Martens Closed Tester, D 93-79,1979.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, November 2004, Pensky-Martens Closed-Cup Method for Determining Ignitability, Method 1010A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1.1, 2018

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

LIST OF TABLES AND FIGURES

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Flashpoint Pensky-Martens Closed-Cup, Method 1010A	Method blank	One per prep batch	No flash	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank
	Sample Duplicate	One sample duplicate per ten samples	Results of sample and sample duplicate agree within ±2 °C – Report the lowest value.	(1) If lab QC in criteria and duplicates do not agree within ±2 °C , report the lowest value and narrate the other values. (2) Else, reanalyze
	LCS / p-xylene	In duplicate per batch of twenty samples or less	Flash point 27°C ± 2°C	1)Repeat analysis of reference standard and associated samples

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-736-07	METHOD 1010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

FIGURE 1

EXAMPLE OF FLASHPOINT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC. - FLASHPOINT – CLOSED CUP LOGBOOK

WG107342 R153375

METHOD: SW 846 1010	p-XYLENE: 27 Degrees Celcius	Ambient Barometric Pressure (*) = 749.3 mm Hg (in. of Hg) X (25.4 mm/in) 29.50
PQL: 25 Degrees Celcius	p-XYLENE Lot Number: SWL3203	

Sample ID	Analysis Time	Start Temperature (°C)	End Temperature (°C)	Flash? (Check appropriate box & record temp.) YES	Temp.(°C)	NO	Reported Flashpoint Corrected For Ambient Barometric Pressure	Comments
p-Xylene (LCS)	13:05	25.0	25.0	✓	25.0		25.353	
p-Xylene (LCS)	13:10	25.0	27.0	✓	27.0		27.353	
Method Blank	13:27	25.0	71.0			✓		
SF2013-3	13:45	25.0	71.0			✓		
-3Dup	15:00	25.0	71.0			✓		
SF2151-4	15:19	25.0	71.0			✓		
SF2152-4	15:35	25.0	71.0			✓		
			DW	4/24/12				

QAWL633

* = Ambient Pressure of the laboratory at the time of the test. When the pressure differs from 760 mm Hg, correct the flashpoint as follows: Corrected Flashpoint = (Observed Flashpoint in °C) + 0.033 (760 - the ambient barometric pressure in mm Hg) .
0.353

Analyst DW Date 4/24/12
Reviewed By [signature] Date 04/24/12

WL-029 – REVISION 1 – 10/06/2010

0000022

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

FIGURE 2

BATCH SHEET FOR FLASHPOINT

WET CHEMISTRY BATCH REPORT
Apr 24 2012, 03:49 pm
Batch: WG107342

Parameter: Ignitability
Date Analyzed: 24-APR-12
Analyst Initials: DW

Prep Date: N/A
Prep Method: N/A
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF2013-3	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71.	71.		
SF2151-4	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71.	71.		
SF2152-4	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71.	71.		
WG107342-1	LCS	SW846 1010	1.0000mL	1.0000mL	1	25.353	25. Deg. C	NA	71	71.	71.		94
WG107342-2	LCSD	SW846 1010	1.0000mL	1.0000mL	1	27.353	27. Deg. C	NA	71	71.	71.	8	101
WG107342-3	MBLANK	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71.	71.		
WG107342-4	DUP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71.	71.	NC	

Comments:

WG107342-1	SF2013-3
WG107342-2	SF2013-3
WG107342-3	SF2013-3
WG107342-4	SF2013-3

Entered by: DW Date: 4/24/12 Accepted by: [signature] Date: 04/24/12

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: BP

Review Date: 1-10-20

SOP Number: CA-736-07

SOP Title: Test Method for Flash-Point By Pensky-Martens Closed Cup tester

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 01/13/20

QAO Signature: _____ **Date:** 01.13.20

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

Prepared By: Greg Lull Date: 7/2002

Approved By:

Group Supervisor: Keith Tanguay Date: 091102

Lab Operations Mgr: Jol C. Butler Date: 9/11/02

QA Officer: Deborah J. Nadeau Date: 9.11.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. start up, added TS to calc., removed steps not currently in practice. Sect. 8 - updated LCS and CCV information. Updated Table 1	LAD	01/07	01/07
02	Added method SW846 9060 to title and Table 2. Added 9060 quadruplicate Sample analysis to sect. 7 and Table 1. Removed D.I. water from MB definition. 5.3-added TIC std. info. Sect. 7.0 - rewrote for clarity on instrument and software instructions. Fixed typos- sect. 2.	LAD	06/08	06/08
03	Minor changes to reflect current equipment, practices and techniques.	LAD	08/09	08/09
04	Added 9060 QC requirements to Table 1. Added method modifications to Table 2. Minor edits to Section 7 to reflect current practice, remove redundancy and for clarification. Updated and/or added references to Section 7.9 and 10.	LAD	06/10	06/10
05	Removed 1:1 phosphoric acid and added 1:1 hydrochloric acid. Added MDL, LOD and LOQ information. Updated Figures 1 → 4.	LAD	02/13	02/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 5 – Updated for current CCV and LCS source and preparation.	LAD	05/16	05/16
07	Added Particulate Organic Carbon to Sect. 1&2. Sect. 8 - Added contingency plan. Added Attachment A - preparation and calculation of particulate organic carbon. (POC)	LAD	05/18	05/18
08	Sect. 1 - Added muffled sand to M. Blk and CCB definitions Sect 6 - Added 0.5M HCl to TOC calibration std. prep., Added muffled sand and the preparation. Sect. 9 - Added requirement to dry M. Blk, LCS and CCVs. Updated Logbook Ex.	LAD	01/19	01/19

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

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I acknowledge receipt of copy ___ of document SOP **CA-741-08**, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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, titled **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services technical personnel to determine Total Organic Carbon (TOC) in solids and Particulate Organic Carbon (POC) in aqueous matrices in accordance with EPA Region II Lloyd Kahn method and SW846 9060 Mod.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 µg/g.

This method may also be used to determine the Particulate Organic Carbon (POC) associated with aqueous samples. The preparation and calculation of POC is detailed in Attachment A of this SOP.

1.1 Definitions/Acronyms

TC – Total carbon

IC – Inorganic Carbon

TOC – Total Organic Carbon

POC - Particulate Organic Carbon

Method Blank – A sample boat filled with approximately halfway with muffled sand, no reagents are added and is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is a sample boat filled approximately half way with muffled sand, no reagents are added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

2.0 SUMMARY OF METHOD

Total Carbon (TC) is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon-to-carbon dioxide (CO_2) in the presence of oxygen. The amount of CO_2 derived from a sample is directly proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 hydrochloric acid to the sample and combusting it at 103^{o} C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon.**

To determine Particulate Organic Carbon (POC), an aqueous sample is filtered through a pre-combusted / pre-cleaned glass fiber filter to obtain the particulates on the filter. The filter + particulates sample is then dried. The dried filter + particulates are then milled, ground, and homogenized prior to removal of an aliquot to place in the sampling boat, treated with HCl to remove IC (using the same procedure as for TOC) and placed into the carbonaceous analyzer for combustion at 900^{o}C. Procedures for POC are detailed in Attachment A of this TOC SOP.

3.0 INTERFERENCES

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu model TOC-Vcph with NDIR.

4.2 SSM-5000A 900^{o}C furnace with boat sampling module.

4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.

4.4 Ceramic boats.

4.5 Drying oven capable of maintaining 103-105°C

4.6 Oxygen gas

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

5.0 REAGENTS AND STANDARDS

5.1 TOC Calibration / CCV Standard: D(+) Glucose Reagent ACS Anhydrous, ACROS

5.1.1 Calibration / CCV Solution – Prepare using 25.0 g and bring up to 250 mL with DI water. Before reaching the 250 mL fill line, add 0.5 mL HCl for preservation. Stable for 3 months.

5.1.1.1 Using the table below, each calibration level is prepared by pipetting the appropriate amount of Calibration / CCV Solution into a ceramic boat filled with approximately 0.5 g of sand.

Calibration Level ug C	Weight (mg)	mL of stock
Blank	0	0
200	0.5	0.005
400	1.0	0.01
2000	5.0	0.05
4000	10	0.1
16000	40	0.4
24000	60	0.6

(Upper range limit for TOC is 24,000ugC)

5.1.1.2 The 16000 ug C level is also used as the CCV

5.2 LCS Stock Standard – Dextrose Anhydrous Powder – Prepare by weighing 5.0 and bringing to 50 mL with D.I. Water. Stable for three months.

5.3 TIC Calibration / CCV Standard Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/gC)

Calibration Standards: weigh into ceramic boat.

0.0 mg - Calibration Blank
3.5 mg - 400ugC
17.7 mg - 2000ugC
35.3 mg - 4000ugC
70.7 mg - 8000ugC
212.0 mg - 24,000ugC

(Upper range limit for TIC is 25,000 ugC)

5.4 1:1 Hydrochloric acid / DI water solution

5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

5.6 Muffled Sand – Play sand muffled at 900 ºC for at least 4 hours.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days for Lloyd Kahn and 28 days for SW846 9060.

7.0 PROCEDURES

SET UP AND CALIBRATION

7.1. Turn on TOC-Vcph analyzer, SSM-5000Afurnace, and oxygen supply and connect to the TOC software program.

7.2. Start the TOC-V software program by double clicking the TOC-V icon on the desktop and selecting sample table editor. Enter user name, passwords and press **OK**. Click the “new” icon followed by “sample run”. Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Click the “connect” icon and choose “Settings on PC”. This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 900^{o}C before beginning analysis. The gauges on the soil module for the gas lines should read 150psi and 200psi from right to left. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

7.3. To run a calibration, minimize the sample run. Click the “New” icon followed by “calibration curve”. This activates the calibration curve wizard.

7.4. Choose SSM-5000A next to system by using the pull down menu, the click **NEXT** twice. Type in the file name specifying that it is a curve in the name (i.e toccurve030510), and then click **NEXT**. Using the pull down menu select ug as the units and click **NEXT**.

7.5. A 6-point curve (for either TC or IC) must be run at least every 3 months to verify the calibration. The calibration may also be updated as necessary as demonstrated by failure of the Laboratory Control Sample (LCS) or Continuing Calibration Verification (CCV). To add the points to the curve select “(1)” then click **ADD**. In the highlighted **Conc:** box type in the concentration of the first point (24000), and then press **OK**. Click on the next point “(2)” and **ADD**. Continue with previous steps until all 6-points

have been entered. Click **NEXT** twice and then **FINISH**. Now the curve needs to be run.

7.6. Maximize the sample run and highlight the first row. Select insert then calibration curve from the top menu. Select the calibration curve that was just saved and click **OK**. The calibration curve will be renamed with the date and time (i.e toccal030510, note: name must include the word curve or cal, whichever was not used in the original file name) when the start button is clicked.

7.7. Once all the boxes on the top of the sample run say “Ready”, click the start icon (traffic light) on the menu bar. Once the calibration has been started the instrument will prompt two windows.

7.8. The first will state which point will be running, verify the point and press **OK**. The second window will ask for the exact amount of dextrose that has been weighed out. Enter the amount and click OK. For the last point, the blank point, an empty sample boat is used and 500mg is used for the weight.

7.9. Once entered click **OK** and the instrument will prompt a message to move the sample boat to the measure position.

7.10. Open the TC sample port and insert the boat. Close the port tight and set timer for two (2) minutes. These two minutes ensures that the CO2 that entered the instrument has had enough time to go completely through the detector and not give any false positives. When the timer goes off slide the sample boat to the measurement position.

7.11. Once the instrument is done analyzing the sample, about 4-5 minutes, there will be a message prompt to slide the sample boat to the cooling position. After a few minutes it will then prompt again to move the sample boat back in to the port. As soon as the boat has been pulled all the way back the instrument will automatically start the process for the next point. Repeat steps 7.8 to 7.10 for the rest of the calibration.

7.12. When the entire curve is complete, print the sample report and check the curve and its linearity. The correlation coefficient must be greater than or equal to 0.995. The value of the calibration checks must fall within the control limits (80-120% recovery). If not rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If problem persists, remake the standards and repeat the procedure or perform instrument maintenance. If maintenance is required, record in TOC instrument maintenance notebook.

7.13. Once there is a valid calibration curve, the curve must be saved to the method. Click file, then open. Select method from the pull down menu and select tc method soils. Click the SSM-TC tab and change the calibration to the new one. Click OK. Samples

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are now ready to run. Select "new" file icon on the menu bar and, then click on sample run. Highlight the first row on the sample run. Click the insert sample icon. The parameter box opens, click the method box and select tc method soils. Click next and enter the sample name. Click next twice then finish.

7.14. Using a calibrated analytical balance weigh out 100-500 mg, wet weight, of the sample into a tarred ceramic boat. Using a disposable pipet add 1:1 hydrochloric acid to the sample until there is no more effervescing, usually 2-4 drops. If a sample concentration is greater than 24000 ug/g, a smaller sample amount must be used to achieve a concentration that is within the curve. Samples that require less than 50 mg must be analyzed in quadruplicate and the standard deviations calculated.

For Method Blanks, LCSs and CCVs, 1:1 hydrochloric acid is not needed.

For TC or Percent TC analysis, 1:1 hydrochloric acid is not needed.

NOTE: Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling

NOTE: Samples analyzed by method SW846 9060 must be analyzed in quadruplicate.

7.15. Dry the samples, Method Blanks, LCSs and CCVs in a 103° - 105° C oven for ten minutes. This process removes the inorganic carbon and residual moisture from the samples.

7.16. To start the analysis, click the start icon. The first sample entered will bring up a file name box. Name the file by analysis and date, click OK. Then the box to enter the weight will open. Enter weight and press OK. Follow steps 7.10 and 7.11 with the exception that each sample is now entered separately throughout the batch.

7.17. Calculate the TC or IC (whichever is being preformed) concentration using the following equation:

7.18.

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

7.18. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter "LLOYD", "Percent", or "TOC" in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file menu. Select "save as" and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.\

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7.19. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Wet Chemistry supervisor or other qualified designee.

7.20. Analysts file printouts of instrument calibrations and sample data in the lab for approximately 3 months for reference. Prior calibrations are archived and all are available for retrieval**.**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A Method Blank is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter and at the end of the run. The Method Blank and CCBs are ceramic boats filled approximately halfway with muffled sand. Results must not be greater than the reporting limit (PQL). The PQL for this test is 400mg/kg.

8.2 Analyze an LCS (40-50 mg of Dextrose = 16000-20000ugC for TC) / (28 mg Sodium Hydogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.

8.3 Run a CCV (40-50 mg Dextrose = 16000-20000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value.

8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. Run a matrix spike duplicate every 10 samples for SW 9060 samples. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

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Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of EPA SW846 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.
TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January 2017

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

LIST OF TABLES AND FIGURES

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/ICV	One per prep batch of twenty samples (Lloyd Kahn); one per fifteen samples (SW 9060)	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	CCV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples. SW 9060 – all samples are analyzed in quadruplicate	≤ 30% RSD	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike/Matrix spike duplicate	One MS per ten samples(MSD required every 10 samples for SW 9060)	75-125% Recovery ≤ 30% RPD	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL and-or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-741-08	METHOD LLOYD KAHN/SW846 9060
Apparatus/Materials		
Reagents	Dextrose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures	Report one of the duplicate samples using an RPD of 30%.	Report average and range of quadruplicate analysis (SW 9060) Use 3 standard deviation limit (Lloyd Kahn)
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

R447211
WG234890

Katahdin Analytical Services

Carbon Analysis of Solid Samples - Shimadzu TOC-V_{CPH} / SSM-5000A

Analysis Type and Method (Check One)	
___ Total Carbon (SW846 9060M)	___ Total Inorganic Carbon (SW846 9060M)
___ Total Organic Carbon (SW846 9050)	___ Other (Specify):
✓ Total Organic Carbon (Lloyd Kahn)	___ Percent Carbon (SW846 9060M)

Spiking Information		Calibration Information	
LCS Spike Source ID / Compound: WI16776	TV: 16000ug	Calibration Date:	8/18/18
CCV Spike Source ID / Compound: WI16817	TV: 1	Calibration Analyst:	SC
MS Spike Source ID / Compound: WI16776	TV: 4000 ug	Calibration Source ID:	WI16817
Acid Lot Number: WI16673		Pipette IDs:	07, 08

Katahdin Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)	% Recovery
CCV	40	(Wet) Dry	98%
CCB	500	(Wet) Dry	
LCS	40	(Wet) Dry	106%
LCSD	40	(Wet) Dry	108%
SL7696-28	242.7	Wet (Dry)	
-33	240.9	Wet (Dry)	
-34	258.6	Wet (Dry)	
-35	258.0	Wet (Dry)	
SL7786-1	279.3	Wet (Dry)	
-2	258.8	Wet (Dry)	
-3	265.5	Wet (Dry)	
-4	328.6	Wet (Dry)	
CCV	40	(Wet) Dry	103%
CCB	500	(Wet) Dry	
SL7786-5	301.0	Wet (Dry)	
-6	283.0	Wet (Dry)	
-7	272.0	Wet (Dry)	
-8	273.5	Wet (Dry)	
-9	250.2	Wet (Dry)	
CCV	40	(Wet) Dry	103%
CCB	500	(Wet) Dry	

* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample. "TV" = True Value

Analyst: ZF	Analysis Date: 8/19/18
Reviewer:	Review Date:

WL-019- Revision 4 – 06/10/2017 QAWL902 0000017

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FIGURE 2

EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Type	Analysis	Sample Name	Dilut	Result	Comment	Status	Date / Time
1	Unknown	SSM-TC	CCV	1.00	SSM-TC:15907 ug		Completed	01/23/13 09:48:06 A
2	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 09:55:01 A
3	Unknown	SSM-TC	LCS	1.00	SSM-TC:15615 ug		Completed	01/23/13 10:10:11 A
4	Unknown	SSM-TC	LCSD	1.00	SSM-TC:15254 ug		Completed	01/23/13 10:18:37 A
5	Unknown	SSM-TC	SG0288-1	1.00	SSM-TC:7567 ug		Completed	01/23/13 10:30:11 A
6	Unknown	SSM-TC	SG0421-1	1.00	SSM-TC:10697 ug		Completed	01/23/13 10:38:56 A
7	Unknown	SSM-TC	SG0421-2	1.00	SSM-TC:8713 ug		Completed	01/23/13 11:49:06 A
8	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6723 ug		Completed	01/23/13 12:07:23 P
9	Unknown	SSM-TC	SG0365-1	1.00	SSM-TC:6614 ug		Completed	01/23/13 12:18:03 P
10	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4359 ug		Completed	01/23/13 12:27:49 P
11	Unknown	SSM-TC	SG0365-6	1.00	SSM-TC:4475 ug		Completed	01/23/13 12:43:35 P
12	Unknown	SSM-TC	SG0443-1	1.00	SSM-TC:13287 ug		Completed	01/23/13 12:55:28 P
13	Unknown	SSM-TC	CCV	1.00	SSM-TC:15752 ug		Completed	01/23/13 01:07:03 P
14	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:14:02 P
15	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:0.000 ug		Completed	01/23/13 01:31:39 P
16	Unknown	SSM-TC	SG0365-11	1.00	SSM-TC:255.1 ug		Completed	01/23/13 01:43:54 P
17	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:6585 ug		Completed	01/23/13 01:52:34 P
18	Unknown	SSM-TC	SG0365-15	1.00	SSM-TC:7112 ug		Completed	01/23/13 02:05:11 P
19	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:7023 ug		Completed	01/23/13 02:19:50 P
20	Unknown	SSM-TC	SG0365-20	1.00	SSM-TC:6512 ug		Completed	01/23/13 02:29:21 P
21	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:11013 ug		Completed	01/23/13 02:41:57 P
22	Unknown	SSM-TC	SG0365-20MS	1.00	SSM-TC:9743 ug		Completed	01/23/13 02:54:30 P
23	Unknown	SSM-TC	SG0473-1	1.00	SSM-TC:105.2 ug		Completed	01/23/13 03:01:58 P
24	Unknown	SSM-TC	SG0473-1MS	1.00	SSM-TC:4586 ug		Completed	01/23/13 03:16:10 P
25	Unknown	SSM-TC	CCV	1.00	SSM-TC:14992 ug		Completed	01/23/13 03:26:56 P
26	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 03:35:06 P
27	Unknown	SSM-TC	SF0473-1MSD	1.00	SSM-TC:5927 ug		Completed	01/23/13 03:48:30 P
28	Unknown	SSM-TC	SG0488-2	1.00	SSM-TC:5709 ug		Completed	01/23/13 03:58:13 P
29	Unknown	SSM-TC	CCV	1.00	SSM-TC:15477 ug		Completed	01/23/13 04:13:04 P
30	Unknown	SSM-TC	CCB	1.00	SSM-TC:0.000 ug		Completed	01/23/13 04:21:43 P

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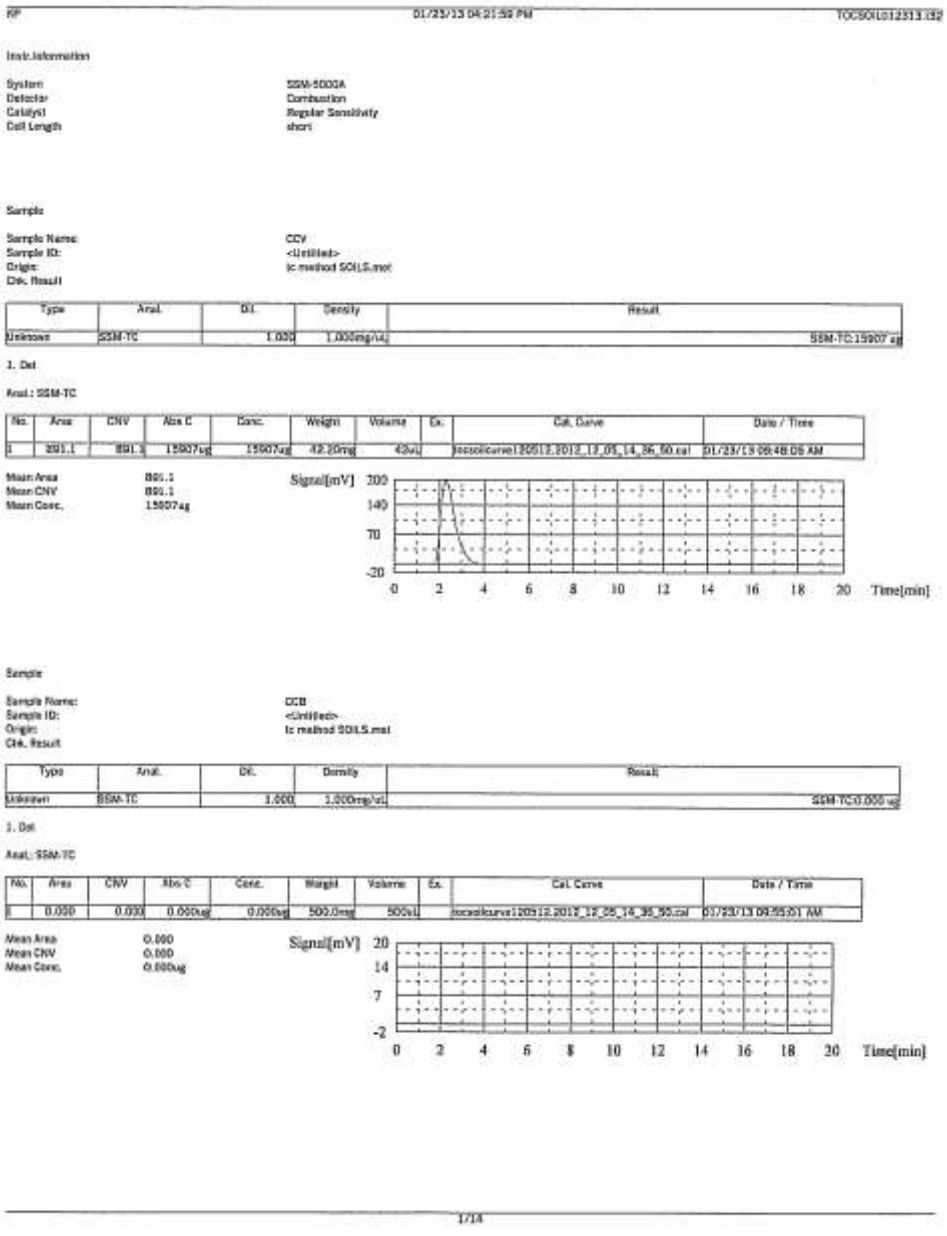
FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT



RP 01/23/13 04:21:58 PM TOC80\LO12313.C02

Instr.Information

System SSM-5000A
Detector Combustion
Catalyst Regular Sensitivity
Cell Length short

Sample

Sample Name: CCV
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:15907 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	891.1	891.1	15907ug	15907ug	42.20mg	42uL		tocsolicurve120312.2012_12_05_14_36_50.cal	01/23/13 09:48:05 AM

Mean Area 891.1
Mean CNV 891.1
Mean Conc. 15907ug

Sample

Sample Name: CCB
Sample ID: <Untitled>
Origin: tc method SOILS.met
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:0.000 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	0.000	0.000	0.000ug	0.000ug	500.0mg	500uL		tocsolicurve120312.2012_12_05_14_36_50.cal	01/23/13 09:55:01 AM

Mean Area 0.000
Mean CNV 0.000
Mean Conc. 0.000ug

1/14

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

FIGURE 4

EXAMPLE OF TOC SOLIDS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Jan 31 2013, 05:05 am
Batch: WG115373

Parameter: TOC In Soil
Prep Date: N/A

Date Analyzed: 23-JAN-13
Prep Method: N/A

Analyst Initials: XP
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SG0443-1	SAMP	SW846 MD06D	141.9mg	200.00ug	1	289215.35693	290000 ug/gdrywt	33.	600	260	1200		
SG0473-1	SAMP	SW846 MD06D	315.4mg	200.00ug	1	414.9499	0500 ug/gdrywt	89.	400	100	500		
WG115373-1	MBLANK	SW846 MD06D	500mg	200.00ug	1	0	0300 ug/gdrywt	NA	400	85.	400		
WG115373-2	LCS	SW846 MD06D	42mg	200.00ug	1	371788.71438	370000 ug/gdrywt	NA	600	85.	400		93
WG115373-3	LCSD	SW846 MD06D	40.6mg	200.00ug	1	375714.28571	380000 ug/gdrywt	NA	600	85.	400	1	96
WG115373-4	MS	SW846 MD06D	264.9mg	200.00ug	1	21537.48061	22000 ug/gdrywt	NA	400	100	500		132
WG115373-5	MSD	SW846 MD06D	282.7mg	200.00ug	1	26092.56574	26000 ug/gdrywt	NA	400	100	500		116

Comments:

SG0473-1	MS/MSD: RE15303-SOIL-011413-726-727
WG115373-1	SG0472-1
WG115373-2	SG0473-1
WG115373-3	SG0473-1
WG115373-4	SG0473-1
WG115373-5	SG0473-1

Entered by: [signature] Date: 1/31/13 Accepted by: [signature] Date: 01/31/13

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

ATTACHMENT A

PARTICULATE ORGANIC CARBON (POC) PREPARATION AND CALCULATION

The following are the additional apparatus, materials, reagents, sample handling, procedures and quality control (in addition to those previously listed for TOC) required for preparing aqueous samples for particulate organic carbon.

4.0 APPARATUS AND MATERIALS

4.1 Oven - capable of maintaining a temperature of 104°C.

4.2 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.3 Glass fiber filter discs without organic binders, 142 mm diameter, nominal pore size 0.7 um. Pre-combusted / pre-cleaned.

4.4 Analytical balance - capable of weighing to 0.1 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 100g, 50g, 5g, 2g, 0.2g

4.5 Spex Certiprep 8000 Mixer/Mill or equivalent (Ceramic Ball Mixer)

4.6 Desiccator with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)

4.7 Graduated cylinder – 1000 mLs.

4.8 Aluminum tins to hold filters for drying

5.0 REAGENTS AND STANDARDS

5.1 Analyte-free water: Water that has been treated to remove organic carbon

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

For this POC method, holding time for aqueous samples has been established as 48 hours from time sampled to time filtered. The analytical hold time of the prepared (dried/homogenized) particulate sample is 28 days from date prepared to date analyzed.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

This holding time can be extended to one year by freezing the prepared/dried POC sample at < 10°C. Holding time is arrested as long as the sample is maintained frozen.

Note: A project may require analysis of Total Suspended Solids (TSS) and Dissolved Organic Carbon (DOC) on the same parent sample as for POC. In such cases, TSS is analyzed first (using pre-combusted / pre-cleaned filter), then the filter + particulates generated during TSS are prepared and analyzed using the procedures in this SOP Attachment A. The Filtrate is acidified and used for DOC analysis.

7.0 PROCEDURES

7.1 Filter Preparation

7.1.1 Pre-combust filters at 500 ºC

7.1.2 Cool combusted filters to room temperature, in a desiccator

7.1.3 The filters are then weighed to 0.0001g. Record the dried filter weight on a small piece of paper. The filters are placed back into their original box with the weight placed on top of the filter. The box of filters must be stored in a desiccator until the time of use

7.2 Assembly filter apparatus

7.3 Place the filter on the support screen and secure.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.4 Record the weight of the prepared filter in the Special Project TSS logbook

7.5 Measure the volume of sample using a graduated cylinder. Typically, the sample volume will be 4L, 1L or 500mL. For POC analysis on aqueous samples, note that for some projects, the entire sample bottle volume must be filtered.

7.6 Quantitatively transfer the sample to the filter holder.

7.7 Gradually apply gentle pressure of 10-30 psi until sample moves through the filter. Collect the filtrate in a 2L polyethylene container.

7.8 When the pressurizing gas begins to move through the filter, or when the liquid flow has decreased at 30 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration. If the project requires the entire sample volume to be used for POC analysis, multiple filters may need to be used to filter the entire sample.

7.10 Rinse the filter apparatus, graduated cylinder and filter 3 times with 50 mL aliquots of filtrate. If DOC is to be measured on the filtrate, fill 3 40 mL VOA vials preserved with H_2SO_4. This MUST be done before any rinses with analyte free water.

7.11 Rinse the filter 3 times with 100 mL aliquots of analyte-free water (this step is especially important for saline samples, where salts may be retained on the filter and affect the final filter weights).

7.12 <u>The material retained on the filter is defined as the POC.</u>

7.13 Filters are placed in an aluminum weighing dish and dried overnight at 104 ºC.

7.14 Cool the filters in a desiccator to room temperature and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5mg or 4%, whichever is less. Record the weight in the Special Project TSS logbook.

7.15 Using a ceramic ball mill, grind filter and the POC retained on the filter. Homogenize sample.

7.16 Remove an approximately 0.5g aliquot of the dried, pulverized, homogenized sample and place in sample boat, treat with several drops of HCl solution to remove inorganic carbon (IC) and analyze for organic carbon as for the Lloyd Kahn Method (see TOC method procedure for analysis details).

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II METHOD LLOYD KAHN AND SW846 9060 MOD.

7.17 Calculations:

$$\frac{\mu g\ C}{\text{Analysis Aliquot (mg)}} * \frac{\text{Total wt mg (Sample + Residue)}}{\text{Residue wt (mg)}} * \frac{\text{Residue wt (g)}}{\text{Vol. Filtered (L)}} = \text{POC } \mu g/L$$

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 A filter Blank is prepared daily for each batch of ≤ 20 samples filtered using 1 L of analyte-free water, or project-specified volume of analyte-free water, and processed in the same manner as the samples. Filter blank result should be non-detect at the sample QL (QL dependent on sample volume).

8.2 A Certified Reference Material (CRM) should be analyzed with the POC at a frequency of 1 per batch of ≤ 20 samples. Acceptance limits are ± 30% of certified value.

8.3 Additional method QC as per TOC method including Method Blank, LCS, MS/MSD or MS/LR, and CCV with same acceptance criteria as TOC (see Table 1 of TOC SOP). Quadruplicate analyses not required for POC. MS/MSD or MS/Lab replicate (LR) frequency 1 per 20 samples.

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: Jeff C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 & 7.20. Complete rewrite of sections 7.11 & 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure & Logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA Soil Freezer Storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed figures 8 and 10. Updated Table and figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes. Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs & a section on retention of samples.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
09	Added new login information for bottle IDS & screen attachment. Added procedures for bar code scanning for internal custody & deleted manual forms. Added new Incoming from KIMS & deleted old. Added form controlled forms to figures.	DR	9.24.10	09/10
10	Sect. 7 - Removed F9 function in printing labels, fixed how the lab ID appears on labels, and fixed how the date needs to be entered. Updated Fig.s 6 and 13.	LAD	08/13	08/13
11	Sect. 7. Updated WC shorts and rushes from log-book to Google Docs, updated microbiological/ food login process, updated bottle labeling. Updated Table 1 and Figures 1 > 7 & 12. Added Figure 17 - Sample Acceptance Policy	LAD	05/16	05/16
12	Sect. 7.15.4 - Added additional fields in login Info. Updated Table 1 Sect. 7.20 - changed time field after preservation and before analyses from 16 to 24	LAB	09/17	09/17
13	Updated Figure 7 - Sample Receipt Condition Report	LAD	01/19	01/19

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-13**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-13**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., President, Department Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified, Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the QAO or President when warranted.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards,” current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS AND STANDARDS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 5). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "rush turn around" samples or "short hold-time" analyses. Notify the appropriate department Managers/Supervisors of these analyses.

7.4.1 Short hold-time analyses need to be entered into the "Wet Chemistry Shorts Spreadsheet" (Figure 6) on the company Google Docs system. Be sure to list the client, number of samples and date and time of the earliest sample.

7.4.2 GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion.

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7.4.3 Notify all applicable personnel of samples with limited hold-time remaining or rush turn around samples. Appropriate supervisors and PMs must be emailed when a client has requested rush results. The email should include the work order number, the client, the matrix, number of samples, analysis requested

and the turnaround time. Samples for microbiology lab should be brought to them immediately.

7.4.4 Parameters that we routinely analyze which have short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 7). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 8 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.11 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.12 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Reports (generated from KIMS), quotes, memos, etc. These forms may provide details on specific client requests. The Incoming will provide information on specifics for log-in. Refer to Figure 11 for an example.

7.13 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.14 Samples requiring microbiological and/or food analyses are stored in the F/M laboratory walkin. For environmental tests, samples are logged in by the sample receipt department and a copy of the chain of custody is brought with the samples. For non-environmental microbiological tests, a workorder number is assigned by

sample receipt but the samples are not logged in. The workorder number, the chain of custody and a copy of the chain of custody are delivered with the samples. The samples are then logged in by the F/M staff. Sample that require both environmental and non-environmental microbiological analyses are usually processed the same as non-environmental samples

7.15 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 12) is generated for the samples received:

7.15.1 Log onto KIMS by entering employee ID under “Username”, employee specific password under “Password” and KIMS under “Database”.

7.15.2 Once logged onto KIMS select “Sample Management” and then “Login”.

7.15.3 Select “New” and the next available Login ID number will automatically be entered. Select “OK” and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select “change” and type in the appropriate number to access the information.

7.15.4 In the Sample Definition Screen, enter the following information.

Top Section of Screen:

Client ID - Enter client sample description.

ReceiveDate - Enter in date that samples were received in the lab in the format Day-Month-Year (ex. August 23, 2013 is 23-AUG-13).

CollectDate - Enter in date that samples were collected in the format Day-Month-YearTime (ex. 8:30am August 23, 2013 is 23-AUG-13).

TAT - Enter TAT for hardcopy report.

DueDate - Due date will automatically be calculated based on calendar days.

VerbalDate - Manually type in verbal due date.
QuoteRef - Enter quote number if applicable.

Project - Enter project number if applicable.

Account - Enter client specific account number.

Account Name - Account name will automatically be entered.

Collected By - Enter name/initials of sampler listed on COC. If unknown, enter “Client”.

Locator - May be used for client ID information when requested by the project manager.

Site - Enter project site name.

Description - May be used for food descriptions.

Discount - No entry-not currently used.

Priority - No entry-not currently used.

Fact. - No entry-not currently used.

Expected - No entry-not currently used.

Mailed - Data Management will enter the mailed date of the report or SDG right after the report is mailed.

Comments - Enter MS/MSD, verbal due date and any sample irregularities if applicable. Also may be used for long client IDs when requested by the project manager.

OrderDate - Current date is automatically entered.

Middle Section of Screen:

Highlight the first sample in the top section of the screen and then proceed with entries in the middle section of the screen.

Matrix - Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code -	Enter analysis code per test requested on COC. Log-in personnel should refer to Project Incomings, quotes or past Work Orders to aid in the entry of correct product codes.
Type -	Product code type will automatically be entered where S = Stand alone P = Parent C = Children
Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Container Type -	Container type will automatically be entered. Please change from the various choices if the automatic entry is not correct. This is especially important for volatiles in soil since there are many types of preservations.
Container Key-	Make sure “Container Type” is populated. Determine how many bottles there are for each container type. Assign bottles by entering sequential letters for each bottle. For example, sample 1 has six containers, one for metals which we’ll assign container ID, “A”, two for PCBs which we’ll assign container IDs, “B” and “C”, and three for volatiles which we’ll assign container IDs, “D”, “E”, and “F”. The letters should be typed in all in a row with no commas or spaces in between. If 26 bottles per samplenum are exceeded the next ‘key’ would be, ‘A1’, ‘B1’ etc. If no container IDs are needed (i.e. for food or field) it is okay to leave the container key field blank. After the Container Keys are entered click ‘SAVE’. This will create the containers section in the bottom section of the screen. This will also initiate the creation of container labels.

Bottom Section of Screen:

Container # - The container ID numbers will automatically fill in for each analysis from the container key information above.

Container Type - The container types will automatically fill in for each analysis from the container key information above.

Current Location - The current location is automatically entered based on the analysis.

Cooler - Currently not used.

pH - Currently not used.

Temperature - Currently not used.

Seal - Currently not used.

Properly Preserved - Currently not used.

Comments - Comments on individual containers may be entered here, i.e. bubble in VOA vial. Comments regarding problems or breaks with internal custody scanning of bar codes are also automatically entered here.

Select Login Info tab at top of screen and proceed with entry:

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager - Initials of Katahdin person overseeing the project.

Client Project Manager – Name of client manager

Contract – Name or number of contract for the job.

Client PO# - Client purchase order.

Project - Project name.
Cooler Temperature - Temperature blanks or cooler temps.

Delivery Services - Method of delivery to the lab.

QC Level - QC Level of report

SDG ID - Sample Delivery Group ID if applicable.

SDG Status - Begin, Continue or End.

Analysis Instructions - PM will enter special instructions regarding project.

Report Instructions - PM will enter special instructions regarding project.

Regulatory List - Not used.

EDD Format - Specific KAS EDD format.

Login Initials – Initials of person that logged the work order in.

Check – Check number when client pays at sample delivery

Select “SAVE” and then “CANCEL”.

Addresses - Select “Addresses” and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under “Project/Account” and select the report to contact under “Address Type”. Select the appropriate boxes for report, report CC and invoice CC. Select “SAVE” and then “CLOSE”.

Refer to Figure 13 for a screen snapshot of the log-in process in KIMS. Log-in personnel should also refer to the current revision of Katahdin SOP, SD-918, KIMS Work Order Approval & Dispatching, for further hints on log-in.

7.15.5 To print the login report, select “Reports”, “Login” and “Login COC”. Enter login number under “Login Number”. Select “OK”, “Run Report” and then “Print”.

7.16 To print labels, select “Reports”, “Login” and “Labels”. Enter login number under “Login/Prelogin”, select “Background (IDXL) (this is the default)”. Select “OK” and then “Print”. After labels print out select “Cancel”.

Note: As stated in "container key" above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-1A, SC9001-1B, SC9001-1C

Example: One job containing two client samples with 2 different containers for each:

SC9002-1A, SC9002-1B, SC9002-2A, SC9002-2B

7.17 Print the Label Bottle Reference report (under reports tab) for a cross reference to use during labeling. This report will list the bottle type and products related to each Container ID.

7.18 Remove samples from cooler and place them on the counter. Organize them by site ID, in the order of the chain and then by sample analysis.

7.19 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.20 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation

discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 24 hours after preservation before sample preparation can begin.

7.21 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 9). The samples are filtered by the Metals Group or the Wet Chemistry Group depending on which group requires the filtered samples.

7.21.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.21.2 A vacuum pump is attached.

7.21.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.21.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.21.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.22 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.23 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 10).

7.24 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

40 mL vial, 125 ml plastic bottle and 4 oz jar labels will have to be placed vertically on the sample container instead of the standard horizontal placement. Additionally, label for 2 oz jars must be placed on the cover.

7.25 Scan the containers into the appropriate storage locations using the following steps. Note that non-environmental food samples are not scanned and are taken immediately to the food/microbiology lab for storage.

7.25.1 In KIMS, click on “containers”. This can also be done at the walk-in computer or on the “D” instrument computer in the VOA lab, depending on where you are storing samples.

7.25.2 Click on “transfer/update” then “transfer” and select. This will bring you to the screen where you scan your badge. **NOTE: make sure you keep your badge available for this**. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.25.3 Scan the barcode on your badge.

7.25.4 Pick “log-in”.

7.25.5 Pick “check-in”.

7.25.6 Select the location you are checking into, i.e. walk-in, VOA Walkin, etc.

7.25.7 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen. The program is set so that you can continuously scan each sample without having to click anything on the screen. The samples do not have to be scanned in numerical order.

7.25.8 Hit “done/save”.

7.25.9 Hit “close/cancel”. This will return you to the badge scanning screen.

Note: An internal custody report may currently be printed, per client request, by the MIS department.

7.26 Place samples in their designated storage locations. Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.26.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.26.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.26.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in “VOA Refrigerator 2” located in the Volatiles Laboratory.

7.26.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.26.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous and soil samples in VOA vials (except those which are preserved with D.I. water) are stored in “VOA walkin” in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a “paint can” and stored in the sample receipt walk-in.

7.26.6 Soil samples for volatile organics analyses (VOA) that are preserved with Laboratory Reagent Grade Water are stored in “VOA Freezer 1” in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented through the bar code system with respect to native samples. Internal chain-of-custody for extracts and digestates is documented on hardcopy batch sheets. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.27 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.28 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 14).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.29 When removing or returning a sample from its storage location, it must be scanned in or out using the bar code on the container.

7.29.1 In KIMS, click on “containers”.

7.29.2 Click on “transfer/update” then “transfer” and select.

7.29.3 This will bring you to the screen where you scan your badge. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.29.4 Scan the barcode on your badge.

7.29.5 Pick the department that you are bringing samples to or from.

7.29.6 Pick “check-in” or “check-out”.

7.29.7 For check-in, select the location you are checking into.

7.29.8 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen.

7.29.9 Hit “done/save”.

7.29.10 Hit “close/cancel”. This will return you to the badge scanning screen.

7.30 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 15) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. The Immediate Internal COC Logbook must always be consulted if there is ever a question about internal custody.

7.31 If there is an error (i.e. a sample was checked out, but not checked back, and you are trying to check it out), an error screen will pop up indicating who made the error. Take note of who made the error and click "accept bottle". This will allow you to continue, and a note will automatically be applied to the record. If you notice somebody making a lot of errors, please talk to them or let a manager know.

7.32 For samples that are consumed during analysis or preparation, i.e. extractables – either log the samples out and then rescan your badge and log them back in to "consumed" or remove the labels in the lab (when finished) and stick them to your lab coat and then return to scan them into "consumed".

7.33 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and rescan back in using the steps in 7.23.

7.34 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated quarterly against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Environmental Health & Safety Manual, current revision.

Katahdin QA Manual, current revision

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

LIST OF TABLES & FIGURES

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES - AQUEOUS					
Acidity	SM 2310B, 305.1	100 mL	P,G	1,2	14 days
Alkalinity- Titrimetric	SM2320B, 310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1/350.2 SM4500NH3 B&H	100 mL	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, SM4500NH3 H	100 mL	P,G	1,3	28 days
Anions (F, Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (calculation from alkalinity)	SM4500-CO2 D				
Biochemical Oxygen Demand-Carbonaceous	SM 5210B, 405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	SM 5210B, 405.1	1 L	P,G	1	48 hours
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	SM4500-Cl E, 325.2	100 mL	P,G	1	28 days
Chlorine, Total Residual	SM4500-Cl G, HACH 8167	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	SM2120B, 110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	SM4500CN G, 335.1	100 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.4	100 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	SM4500-O G, 360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1,12	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F B/C, 340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	SM4500F C, 340.2	200 mL	P only	1	28 days
Free CO2	SM4500-CO2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2, SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	SM4500-NO3 F, 353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1, 3 OR 11	28 days
pH (Laboratory)	SM 4500H B 150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	SM4500-P E, 365.2	100 mL	P,G	1	48 hours

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PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	SM 2540C, 160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	SM 2540D, 160.2	1 L	P,G	1	7 days
Solids-Settleable Solids (SS)	SM2540F, 160.5	1 L	P,G	1	48 hours
Solids-Total Solids	SM 2540B, 160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	SM 2540E, 160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	SM2540C/E, 160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance	SM2510B, 120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	ASTM D516-02, 375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	SM4500-S2 F, 376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	SM4500-SO3 B, 377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	SM2130B, 180.1	100 mL	P,G	1	48 hours
Volatile Fatty Acids	SOP CA-776	(2) 40 mL	VOA vial	17	14 days
ELEMENTAL ANALYSES - AQUEOUS					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	16	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES - AQUEOUS					
EDB, DBCP & 1,2,3-TCP	8011 & 504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP EPH	(2) 1000 mL	Amber Glass	1,12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1, 18	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Petroleum Range Organics	FL-PRO	(2) 1000 mL	Amber Glass	1,12	7days/40days

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PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Total Petroleum Hydrocarbons	TX1005	(2) 40 mL	VOA vial	12	14days/14days
Extractable Total Petroleum Hydrocarbons	CT-ETPH	(2) 1000 mL	Amber Glass	1	7days/40days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days
PCB Congeners	8082	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2) 40 mL	VOA vial	1,11	14days
Chloropicrin	8011 Mod.	(2) 40 mL	VOA vial	1,8,9	14 days
HPLC ANALYSES – AQUEOUS					
HPLC-Explosives	8330A/B/ B Mod.	(2) 1000 mL	Amber Glass	1	7days/40days
GC/MS ORGANIC ANALYSES – AQUEOUS					
Acid Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles – Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables & (SIM)	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics & (limited SIM)	8260/8260 SIM	(3) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics	624	(3) 40 mL	VOA vial	1,8,9	14 days(~)
MICROBIOLOGICAL ANALYSES – AQUEOUS					
Coliform, Fecal (wastewater)	SM 9222D	100 mL	P,G	1,6	6 hours
Coliform, Fecal (wastewater)	Colilert-18 w/ Quantitray	100 mL	P,G	1,6	6 hours
Coliform, Total (wastewater)	SM 9222B	100 mL	P,G	1,6	6 hours
Coliform, Total (drinking water)	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total (drinking water)	SM9223B, Colitag	100 mL	P,G	1,6	30 hours
E-coli (wastewater)	SM9213D	100 mL	P,G	1,6	6 hours
E-coli (wastewater)	SM9223B Colilert w/ Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B, SIMPlate	100 mL	P,G	1,6	8 hours for compliance samples, 24 for non-compliance samples

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SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES – SOLID					
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1/350.2 SM4500NH3 B&H mod.	4 oz	Soil Jar	1	28 days (^)
Anions (F, Cl, Br, NO3, NO2, SO4)	9056	4 oz	Soil Jar	1	48hrs to 28 days (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/9056	4 oz	Soil Jar	1	28days (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	SM4500F B/C, 340.2 mod.	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	9056 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Nitrite-Automated Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	28 days (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	9056 mod./365.2	4 oz	Soil Jar	1	48 hrs (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	SM2540 G, current CLP SOW	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9038	4 oz	Soil Jar	1	28 days (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Carbon	9060	4 oz	Soil Jar	1	28 days
Total Organic Carbon	Llyod Kahn	4 oz	Soil Jar	1	14 days
Total Organic Carbon	Walkley Black	4 oz	Soil Jar	1	14 days
ELEMENTAL ANALYSES – SOLID					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS Elements	6020	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES – SOLID					

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PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Extractable Petroleum Hydrocarbons	MADEP EPH	4 oz	Soil Jar	1	14days/40days
Fuel Oil	ME HETL 4.1.25 & 8015 mod.	4 oz	Soil Jar	1	14days/40days
Petroleum Range Hydrocarbons	FL-PRO	4 oz	Soil Jar	1	14days/40days
Total Petroleum Hydrocarbons	TX1005	4 oz	Soil Jar	1	14days/14days
Extracted Total Petroleum Hydrocarbons	CT-ETPH	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17 & 8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2)40 mL	VOA vial	1,13	28days
HPLC ANALYSES – SOLID					
HPLC-Explosives	8330B/B Mod.	4 oz or ISM sample	Soil Jar	1	14days/40days
GC/MS ANALYSES – SOLID					
Acid Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables & (SIM)	8270/8270 SIM	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics – Low Soil (<200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics & (limited SIM)	8260/8260 SIM	(2) 40 mL	VOA Vial	1	14 days
Miscellaneous – SOLID					
Grain Size (sieve and hydrometer)	ASTM D422	8 oz	Soil jar or bag	1	none
RCRA – HAZARDOUS WASTE CHARACACTERIZATION					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311/8260	100 g	Soil Jar	1	14 days/14 days

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TABLE 1

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PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
TCLP Extraction-Semivolatiles	1311/8270	200 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Pesticides & Herbicides	1311/8081 & 8151	400 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Metals	1311/6010/6020	200 g	Soil Jar	1	28 days/180 days
TCLP Extraction-Mercury	1311/7470	200 g	Soil Jar	1	28 days/28 days
GC/MS ANALYSES – AIR					
Volatile Organics	TO-15	(1) 1.4 or 6 L	Canister	16	30 days
Volatile Organics	MA-DEP APH	(1) 1.4 or 6 L	Canister	16	30 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NznAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate
16 = None
17 = benzalkonium chloride
18 = 0.02g ammonium sulfate, 0.02g copper (II) sulfate pentahydrate

~ Hold time for unpreserved samples is 7 days.

^ Because there are no published holding times for Wet Chemistry soil methods, these are only recommended holding times. They are not regulatory.

Project-specific (i.e. CLP, NYSDEC) hold times take precedence over these hold times as appropriate.

For solid samples, please place parameters of the same analytical group (ie. wet chemistry) in the same container whenever possible. In addition, organic and inorganic parameters should be placed in separate containers. Volatile organics should always be placed in organic-free jars. Several 4 oz. soil jars may be needed when numerous parameters are required.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client	Contact	Phone # ( )	Fax # ( )
Address	City	State	Zip Code
Purchase Order #	Proj. Name / No.		Katahdin Quote #
Bill (if different than above)	Address		
Sampler (Print / Sign)			Copies To:

LAB USE ONLY | WORK ORDER #:

KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP°C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE / PRESERVATIVES

*	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
		/												
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF KATAHDIN HOMEOWNER CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Drinking Water Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below) yes no — Compliance samples may need to be received on ice.

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Requested Services

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms, e-coli	Lead (1st draw)	Safety Test – coliform,N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner: Total Coliform/e-coli, Nitrate, Nitrite, Chloride, pH, Hardness, Uranium, Copper, Iron, Lead, Manganese, Sodium, Arsenic
													FHA/MSH: Standard plus Lead(1st draw), Turbidity, Color, Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at temperatures specified in the regulations. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements. All services shall be governed by Katahdin's standard terms and conditions.

QA-059 – Revision 2 – 03/31/2016

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF KATAHDIN FOOD/MICROBIOLOGY CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Food & Microbiological Services
Shipping: UPS Fed-Ex	Airbill No.:		
Temperature:			

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (AHS)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

The terms and conditions on the following page hereof shall govern services, except when a signed contractual agreement exists.

QA-058 – Revision 1 – 09/22/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN AIR CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Air Analysis Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Requested Services
Shipping:	UPS Fed-Ex Mail Drop-Off		

Sample Description (Sample Identification and/or Lot #)	Collection					Matrix	Sampler	Can Size	Can ID	Flow Controller ID				Comments
	Date	Start Time	End Time	Initial Vac	Final Vac									

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Katahdin inspects and verifies all equipment including, but not limited to, canisters and flow controllers before being sent to the client. As the client you have agreed to pay a rental fee for use of this equipment, which is the sole property of Katahdin. All equipment will be inspected for damage and completeness upon return to Katahdin. In the event that rental equipment is missing and/or damaged, by signing this COC, you (the client) agrees to pay Katahdin for replacement of any unuseuable, damaged or missing equipment.

QA-132 – Revision 1 – 02/06/2014

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

KATAHDIN ANALYTICAL SERVICES, LLC.
SAMPLE LOG IN

pH Paper Lot #: FK 564756

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
5/13/16	1345	5-13-16	1400	SJ 3311	Harmon's	
5/13/16	1350	5-13-16	15:00	SJ 3312	Bristol Seafood	
↓	↓			SJ 3313	Camp Sunshine	↓
5-13-16	14:00			SJ 3314	DEP-B	
				SJ 3315	↓	
				SJ 3316	DEP-A	
				SJ 3317		
			15:30	SJ 3318	FGS	
				SJ 3319	CES	
				SJ 3320		
				SJ 3321		
				SJ 3322	SWCole	
		5-13-16	16:00	SJ 3323	Clearwater	
				SJ 3324		
				SJ 3325		
				SJ 3326		
				SJ 3327		
				SJ 3328		
				SJ 3329		
				SJ 3330		
				SJ 3331		
				SJ 3332		
				SJ 3333		
				SJ 3334	PWD	open
		5-13-16	16:40	SJ 3335	Cape Elizabeth Tax	
5-13-16	15:00			SJ 3336	MEL	
↓	↓			SJ 3337	↓	↓
5-13-16	15:35			SJ 3338	PWD	
5/13/16	1540			SJ 3339	Maine Medical	
				SJ 3340	COLM	
				SJ 3341		

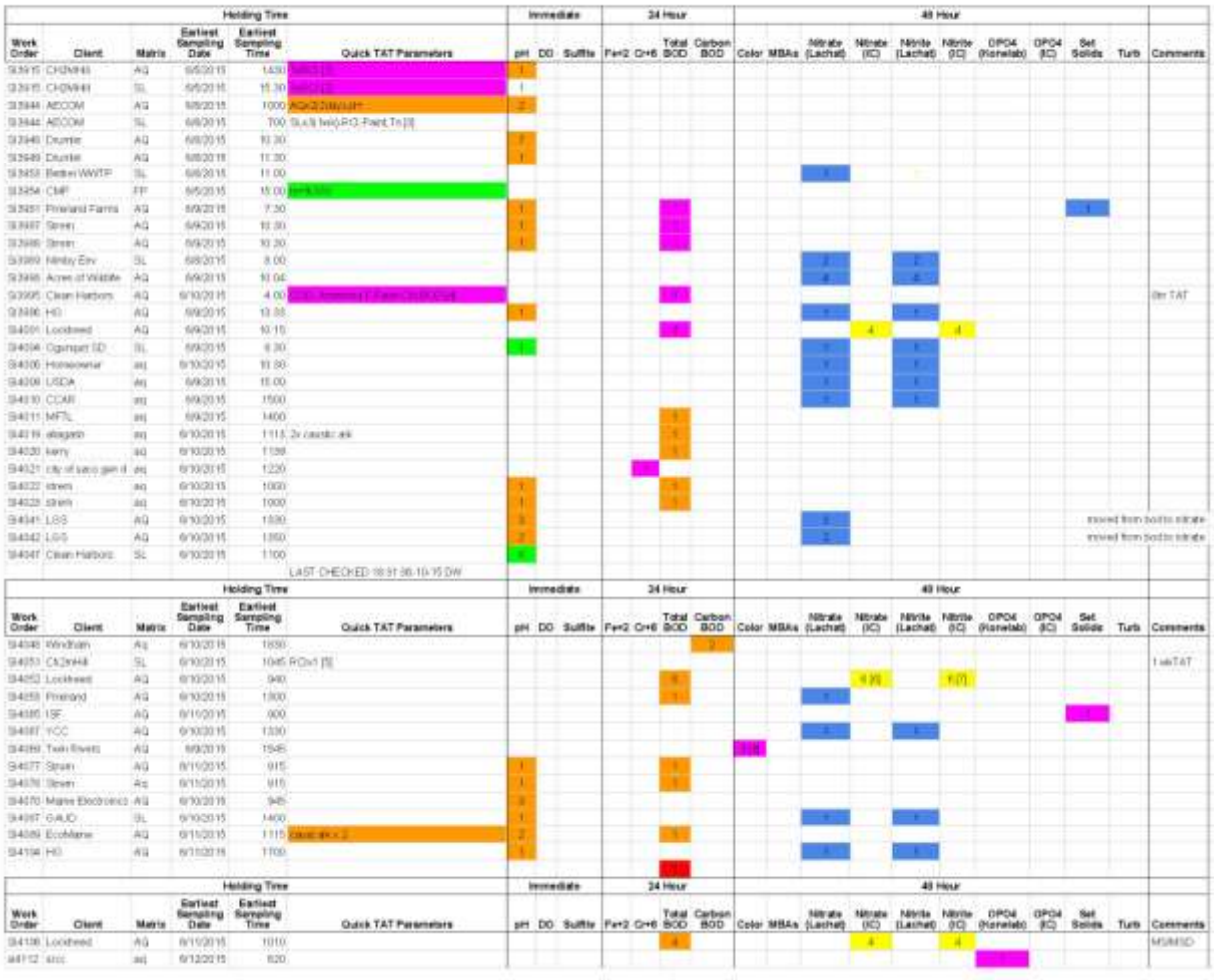
QA-032 - Revision 1 - 12/30/2009 Updated: 04/26/2016

QAQC793 0000011

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES SCREEN SHOT



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, LLC. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C): / Thermometer ID: IR-1
Samples received at <6 °C w/o freezing?					Note: Not required for metals (except Hg soil) analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) or insufficient ice may not meet certain regulatory requirements and may invalidate certain data.
If yes, was there sufficient ice to meet temperature requirements?					
If temp. out, has the cooling process begun (i.e. ice or packs present) and sample collection times <6hrs., but samples are not yet cool?					Note: No cooling process required for metals (except Hg soil) analysis.
6. Volatiles: **Aqueous:** No bubble larger than a pea?					
Soil/Sediment: Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
D.I. Water - Received within 48 hour HT?					
Air: Refer to KAS COC for canister/flow controller requirements	√ if air included				
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					
11. Bottleware Prepped on:					

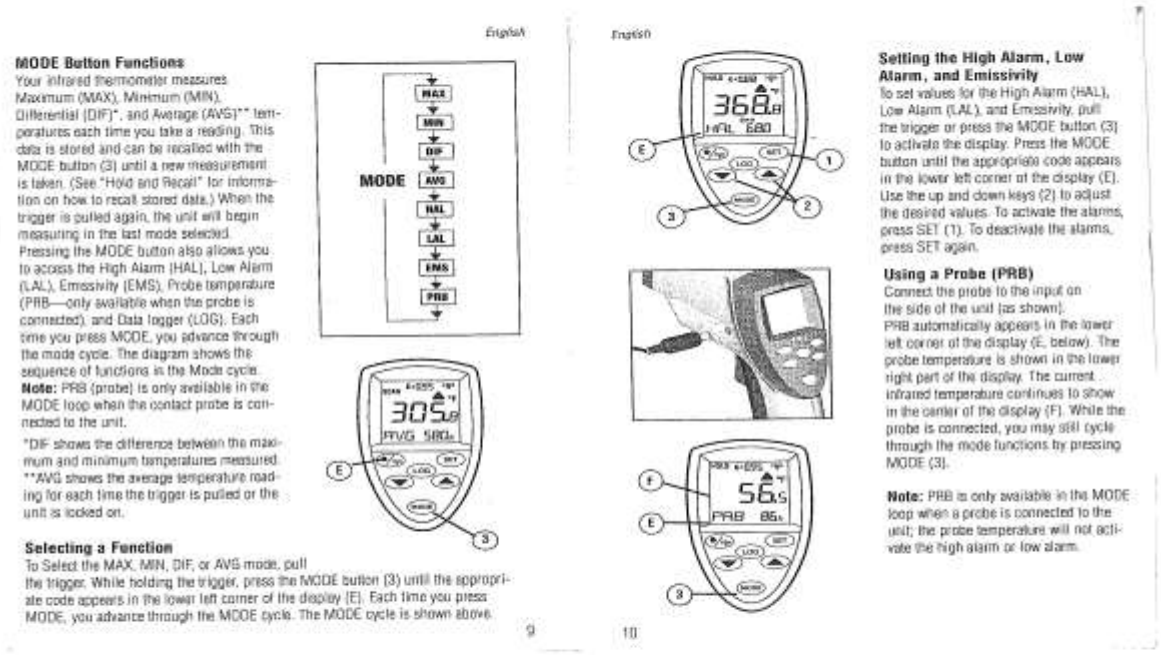
* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments.

QA-048 – Revision 7 – 09/26/2018

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY



English

MODE Button Functions

Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

Selecting a Function

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.

9

English

Setting the High Alarm, Low Alarm, and Emissivity

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

10

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By:		Filtered and Preserved By:		
		Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ **Date:** _____

ME-005 - Revision 1 - 09/23/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst

REVIEWED BY: DATE:

QA-068 – Revision 1 – 09/23/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KIMS LABORATORY INCOMING SAMPLE REPORT

Katahdin ANALYTICAL SERVICES

INCOMING SAMPLE REPORT

Quote: ELM001 Account: ELM001 Project:
Company: [redacted] Quote Date: 29-MAR-10 Expires:
Name: [redacted] Date Expected: 28-SEP-40
Address: [redacted] Email: [redacted]

Notes: 8-aqueous VOA, 4-MEE,22-TOC, 44-metals (Total & Dissolved), 22-alkalinity, 12-Chloride,Nitrate, Nitrite, Sulfate

Analysis Notes: Merge Results for EDD

Report Notes: Email pdf and EDD to maltmayer@elmllc.com and tsnarr@elmllc.com, Mail rpt and EDD on CD, no HC, metals need to be rpt mg/l. Down load rpt to FTP site for ELM. Hold Rpt Till Payment See Daphne

Description:
Project Name: Pilot Test [redacted] Client PO:
QCLevel: II Tat: 13 Terms: Reg List: Edd: KAS064-XLS

Product	Matrix	Quant	STD or Special Lists	Short	Unit Price	Total Price
E325.2-CHLORIDE	AQ	1	STD		40	40
E353.2-NITRATE	AQ	1	STD	SHORT	0	0
E353.2-NITRITE	AQ	1	STD	SHORT	0	0
E375.4-SULFATE	AQ	1	STD		0	0
RSKSOP175-MEE	AQ	1	STD		85	85
SM5310B-TOC	AQ	1	STD		25	25
SW3010-PREP	AQ	1	STD		0	0
SW6010-ARSENIC	AQ	1	STD		60	60
SW6010-ARSENIC-DIS	AQ	1	STD		60	60
SW6010-CALCIUM	AQ	1	STD		0	0
SW6010-CALCIUM-DIS	AQ	1	STD		0	0
SW6010-IRON	AQ	1	STD		0	0
SW6010-IRON-DIS	AQ	1	STD		0	0
SW6010-MAGNESIUM	AQ	1	STD		0	0
SW6010-MAGNESIUM-DIS	AQ	1	STD		0	0
SW6010-MANGANESE	AQ	1	STD		0	0
SW6010-MANGANESE-DIS	AQ	1	STD		0	0
SW6010-POTASSIUM	AQ	1	STD		0	0
SW6010-POTASSIUM-DIS	AQ	1	STD		0	0
SW6010-SODIUM	AQ	1	STD		0	0
SW6010-SODIUM-DIS	AQ	1	STD		0	0
SW8260FULL_LD	AQ	1	STD		115	115
					$	$85.00

History: _____
Other: _____
_____

Printed: 21-SEP-10 1 of 1

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Login Chain of Custody Report (lno1)

Jan. 26, 2007
03:51 PM

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services
Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

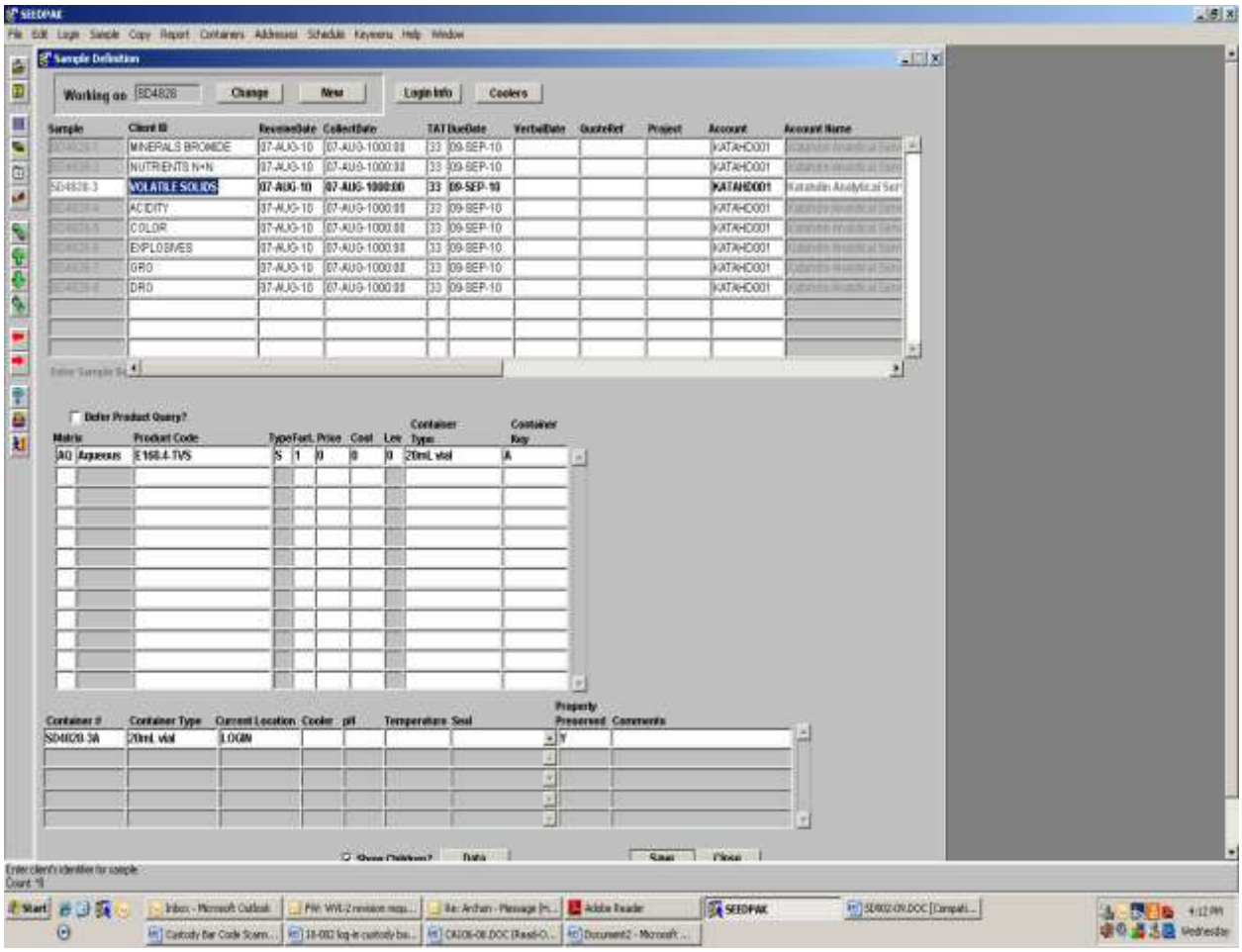
Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	09-FEB-07			3		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	09-FEB-07			3		

Total Samples: **2** ***Total Analyses:*** **2**

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF LOGIN SCREEN IN KIMS



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

Sample Receipt Refrigerator and Freezer Temperature Logbook

Corrective Action: If acceptance criteria are not met, notify the QAO or your supervisor immediately to determine corrective action to be taken. Document the corrective action in the Comments section.

Thermometer Location		Sample Receipt Refrigerator 1	Sample Receipt Freezer 1	Comments
Acceptance Criteria		Above 0 to 6 °C	< -10 °C	
Date	Initials	Temp (°C)	Temp (°C)	

QA-069 – Revision 1 – 06/12/2013

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 15

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

QA-046 – Revision 1 – 04/15/2010

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no

0000001

FIGURE 16

SAMPLE ACCEPTANCE POLICY

Katahdin Analytical Services Sample Acceptance Policy

Katahdin Analytical Services reserves the right to refuse any samples due to any anomalies, discrepancies or non-compliances concerning the receipt and/or analysis of samples. These may include but are not limited to:

- Insufficient sample volume
- Insufficient remaining holding time
- Health or safety risks the samples may pose, including radioactivity
- Insufficient experience to handle sample or analysis
- Improper or illegible labeling of samples
- Improper sample containers
- Insufficient documentation including sample identification, location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample
- Damaged, contaminated or inadequately preserved samples

Any decisions to reject samples are made with the client's input.

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: _____ Date: 2/01

QA Officer: _____ Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution~~ prevention, added updated log-book and greater detail on dis-posal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regu-lations & to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509
05	Sect. 7 - Added non-hazardous samples are recycled, added PCB information, changed elementary neutralization target pH to 5-~~8~~9. Added wording for clarification. Updated Figures 1, 3 and 5.	LMD	06/13	06/13

TITLE: SAMPLE DISPOSAL

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Corrected typos, updated Figure 3	LAD	09/17	09/17

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-06**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-06**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A “Solid Waste” which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined,

to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory President.

It is the responsibility of the Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory President.

It is the responsibility of the Laboratory President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS AND STANDARDS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handling samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles (VOA)
VOA (SL)	Solid Volatiles (VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous liquid samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples and their containers are disposed of with the recycling trash, which is picked up by commercial trash collectors and ultimately turned into construction material.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream “K”. Containers are disposed of with general trash. Solid samples are placed into waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream “A”. Containers are disposed of with general trash. Solid samples are placed in waste stream “L” with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)
Refer to section 7.10 below.

7.9.5 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “Q”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “F” with their containers. The disposal date is recorded in the SDL. Any PCB samples with PCB content 50 ppm or greater, solid or aqueous, are set aside for TCSA regulated disposal.

7.9.6 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “O”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “NHi”. Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.8 Miscellaneous Disposal (as determined by the preservative)

7.9.8.1 Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream “A”. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

7.9.8.2 Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the “O” waste stream. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. Lastly, samples marked “free product” on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium

hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 5 and 9. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Dumping Basic samples (as determined by the basic preservative or by laboratory testing). If the samples have been to be hazardous due solely to the corrosivity characteristic, they are included in the neutralization process above.

7.12 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a “lab pack” of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.13 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Environmental Compliance Officer and the Katahdin President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

QA-022 - Revision 1 - 11/09/2009

QAQC645

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
56-1969-1	M	4-10-13		✓					✓				5-20-13	5-20-13
56-1970-1		4-9-13		✓										
56-1977-1		4-4-13	✓											
56-1978-1-22		4-18-13	1-10, 12-14 16-22		11,15									
56-1979-2-10		4-10-13	✓											
56-1990-1		4-4-13				✓								
56-1991-1	↓	4-4-13				✓							↓	↓
56-1002-1	WC	2-27-13	✓										5-21-13	5-21-13
56-1010-1		2-26-13	✓											
56-1017-1		2-21-13	✓											
56-1021-1		2-26-13	✓											
56-1022-1,2	↓	2-28-13	2	1									↓	↓

0000012

TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value	
SA6605-1		NEED	12/02/07			
SA6606-1		NEED	12/02/07			
SA6607-1		NEED	11/15/07			
SA6608-1		NEED	12/06/07	ORG	1.17	MG/L (HIGH)
SA6608-1		NEED	12/06/07			
SA6608-2		NEED	12/06/07	AA	13	MG/KG (HIGH)
SA6609-1		NEED	11/26/07			
SA6609-1		NEED	11/26/07			
SA6610-1		NEED	11/30/07			
SA6611-1	FCS-020	NEED	12/07/07			
SA6611-2	FCS-020	NEED	12/07/07			
SA6611-3	FCS-020	NEED	12/07/07			
SA6611-4	FCS-020	NEED	12/07/07			
SA6611-5	FCS-020	NEED	12/07/07			
SA6611-6	FCS-020	NEED	12/07/07			
SA6611-7	FCS-020	NEED	12/07/07			
SA6611-8	FCS-020	NEED	12/07/07			
SA6612-1	NSA-030	NEED	12/07/07			
SA6612-2	NSA-030	NEED	12/07/07			
SA6612-3	NSA-030	NEED	12/07/07			
SA6612-4	NSA-030	NEED	12/07/07	ORG	1.70735	MG/L (HIGH)
SA6612-5	NSA-030	NEED	12/07/07	ORG	1.0481	MG/L (HIGH)

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FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Month: _____, 20___

Item / Date:					
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)					
9. Time of Inspection					
10. Verification of Inspection (Name/Date)					

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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0000029

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 5-9-13		Time: 16:30	Analyst: GW
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	7		
5	6		
5	5		
5	7		

Date: 5-16-13		Time: 12:00	Analyst: GW/WS
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	7	good	
5	5		
5	7		
5	7		
6	5		
6	8		
5	7		
5	7		
4	6		

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 1.10.19

SOP Number: SD-903-06

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-11-19

QAO Signature: Leslie Dimond **Date:** 01.24.19

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Ezekiel Lord

Review Date: 01-09-2020

SOP Number: SD - 903 - 06

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 1-9-20

QAO Signature: Lexie Dimond **Date:** 01.10.20