Uniform Federal Policy-
Quality Assurance Project Plan Addendum No. 1
Hazardous Toxic and Radioactive Waste Project
Remedial Investigation
Iona Island Naval Ammunition Depot
Formerly Used Defense Site
Stony Point, Rockland County, New York
(FUDS Project Number C02NY074403)

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	Degrees Celsius
%	Percent
%R	Percent recovery
95UCL	95th Percent upper confidence limit of the mean
ADR	Automated Data Review
AES	Atomic emission spectrometry
Alion	Alion Science and Technology Corporation
AMSL	Above mean sea level
AOC	Area of concern
APP	Accident Prevention Plan
ASTM	ASTM International
AVS	Acid volatile sulfide
B.A.	Bachelor of Arts
BERA	Baseline Ecological Risk Assessment
bgs	Below ground surface
B.S.	Bachelor of Science
CAS	Chemical Abstracts Service
CCB	Continuing calibration blank
CCV	Continuing calibration verification
CEC	Cation-exchange capacity
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHMM	Certified Hazardous Materials Manager
CIH	Certified Industrial Hygienist
COC	Contaminant of concern
COPC	Contaminant of potential concern
COPEC	Contaminant of potential ecological concern
CPR	Cardiopulmonary resuscitation
CSM	Conceptual site model
CSP	Certified Safety Professional
CVAA	Cold vapor atomic absorption
D	Difference
DCS	Duplicate control sample
DER	Division of Environmental Remediation
DERP	Defense Environmental Restoration Program
DoD	Department of Defense
DO	Dissolved oxygen
DQO	Data quality objective

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

DU	Decision unit
EA	EA Engineering, P.C. and its affiliate EA Science and Technology
EcoSSL	Ecological Soil Screening Level
EDMS	Environmental Data Management System
ELAP	Environmental Laboratory Accreditation Program
EOD	Explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
ERA	Ecological risk assessment
ERIS	Environmental Restoration Information System
ERPIMS	Environmental Resources Program Information Management System
FS	Feasibility study
ft	Foot (feet)
FUDS	Formerly Used Defense Site
FUDSChem	Formerly Used Defense Site Online Chemical Database
g	Gram(s)
GC/MS	Gas Chromatography/ Mass spectrometry
GIS	Geographic information system
GPS	Global positioning system
Greeley-Polhemus	Greeley-Polhemus Group, Inc.
H_a	Alternative hypothesis
H_0	Null hypothesis
H2S	Hydrogen sulfide
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High density polyethylene
HHRA	Human health risk assessment
HI	Hazard index
HMW	High molecular weight
HNO_3	Nitric acid
HPLC	High performance liquid chromatography
HQ	Hazard quotient
HTRW	Hazardous toxic and radioactive waste
ICAL	Initial calibration
ICB	Initial calibration blank
ICS	Interfering element check standards
ICS-A	Interference check solution A
ICS AB	Interference check solution AB
ICP	Inductively coupled plasma

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

ICPMS	Inductively coupled plasma mass spectrometry
ICV	Initial calibration verification
IDL	Instrument detection limit
IRP	Installation restoration program
ISM	Incremental sampling methodology
ISO	International Organization for Standardization
in.	Inch(es)
ITRC	Interstate Technology Regulatory Council
kg	Kilogram(s)
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LEP	Licensed Environmental Professional
LMW	Low molecular weight
LOAEL	Lowest-observed-adverse-effect level
LOD	Limit of detection
LOQ	Limit of quantitation
MCL	Maximum contaminant level
MDL	Method detection limit
MEC	Munitions and explosives of concern
mg/kg	Milligram(s) per kilogram
mg/l	Milligrams per liter
mL	Milliliter(s)
MMRP	Military Munitions Response Program
MS	Matrix spike
M.S.	Master of Science
MSA	Measurement systems analysis
MSD	Matrix spike duplicate
NA	Not applicable
NAE	New England District
NCP	National Contingency Plan
ND	Non-detect
No.	Number
NOAEL	No observed adverse effect level
NS	No standard
NRWQC	National Recommended Water Quality Criteria
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

ORP	Oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
oz	Ounce(s)
PAH	Polycyclic aromatic hydrocarbon
PAL	Project action limit
PDF	Portable document format
PDS	Post digestion spike
P.E.	Professional Engineer
P.G.	Professional Geologist
PIPC	Palisades Interstate Park Commission
PM	Project Manager
POC	Point-of-contact
ppm	part per million
PQL	Project quantitation limit
Q-Q	Quantile-quantile
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
QCS	Quality control supervisor
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial investigation
RL	Reporting limit
RPD	Relative percent difference
RSD	Relative standard deviation
RSL	Regional screening level
RSLERA	Refined screening-level ecological risk assessment
SARA	Superfund Amendments and Reauthorization Act
SEDD	Staged electronic data deliverables
SEM	Simultaneously extracted metals
SI	Site Inspection
SIM	Selected ion monitoring
SLERA	Screening-level ecological risk assessment
SM	Standard method
SOP	Standard Operating Procedure
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
STR	Senior technical review

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

SU	Sampling unit
SUXOS	Senior Unexploded Ordnance Supervisor
TAL	Target Analyte List
TBD	To be determined
TM	Technical Manager
TOC	Total organic carbon
TPP	Technical project planning
TSCA	Toxic Substances Control Act
µg/L	Microgram(s) per liter
µmol/g	Micromoles per gram
UFP	Uniform Federal Policy
UPL	Upper Predictive Limit
USACE	U.S. Army Corps of Engineers
USAGC	United States Army Geospatial Center
USCS	Unified soil classification system
USFWS	U.S. Fish and Wildlife Service
UTL	Upper Tolerance Limit
UV	Ultraviolet
UXO	Unexploded ordnance
UXOSO	UXO Safety Officer
V	Verification
VE	Validation electronic
VEM	Validation electronic and manual

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INTRODUCTION

EA Engineering, P.C. and its affiliate EA Science and Technology (EA)[1] has been tasked by the U.S. Army Corps of Engineers (USACE) to complete a Remedial Investigation (RI) at the Iona Island Naval Ammunition Depot Formerly Used Defense Site (FUDS) for the Hazardous Toxic and Radioactive Waste (HTRW) project (FUDS Project Number [No.] C02NY074403). The FUDS is located in Stony Point, Rockland County, New York (Figure I-1). This document is an Addendum to the original Uniform Federal Policy (UFP) - Quality Assurance Project Plan (QAPP) finalized in January 2020 (EA 2020a). The January 2020 UFP-QAPP will hereafter be referred to as the UFP-QAPP (EA 2020a), while this Addendum will be referred to as the UFP-QAPP Addendum.

A two-phased approach (Phase I and Phase II) is being employed during the HTRW RI to facilitate data collection, evaluation, and discussion. The work is being performed by EA under W912DR-15-D-0014 Delivery Order No. W912DR18F0587, under the oversight of the USACE New England and Baltimore Districts. Phase I was conducted in 2020 in accordance with the UFP-QAPP (EA 2020a) and consisted of onsite and background soil sampling and a shoreline reconnaissance along Iona Island. Phase I field activities and analytical results are presented in two approved technical memorandums that were submitted to USACE as follows (Appendix A and Appendix B of this UFP-QAPP Addendum):

- Phase I HTRW Technical Memorandum that was submitted to USACE on 4 September 2020 (Appendix A)
- Iona Island Shoreline Reconnaissance Survey Technical Memorandum that was submitted to USACE on 15 April 2020 (Appendix B).

1.1 PHASE I SUMMARY

Eleven of 32 onsite Phase I Decision Units (DUs) are recommended for no further investigation during Phase II as either analytical results were below EPA screening criteria and/or background concentrations (Figure I-2) or preliminary evaluations of risk did not indicate that concentrations of contaminants of potential concern (COPCs) posed potential risk to terrestrial ecological receptors. Two additional onsite DUs were not sampled during Phase I, including DU-8 due to the presence of debris observed within the DU that was not related to historical DoD operations, and DU-24 due to the lack of soil cover within the DU.

Target Analyte List (TAL) metals and polycyclic aromatic hydrocarbons (PAHs) in soil samples collected 19 onsite Phase I DUs (DU-1 through DU-7, DU-9 through DU-19, and DU-22) sampled during Phase I were reported at concentrations above both EPA screening criteria and background concentrations. These 19 DUs are further discussed in the subsections below.

[1] EA Engineering, P.C. is affiliated with EA Engineering, Science, and Technology, Inc., PBC who does business as EA Science and Technology in State of New York.

1.1.1 DU-1 through DU-7, DU-9 through DU-19, and DU-22: Metals and PAHs in Upland and Hydric Soil

Of the 19 DUs with concentrations above both EPA screening criteria and background concentrations, 16 were established in the upland area of Iona Island where upland soil is present (DU-1 through DU-7, DU-9 through DU-16, and DU-22), and 3 were established within the marsh area between Iona and Round Islands where hydric soil is present (DU-17, DU-18, and DU-19). These two areas (upland and hydric soil) are discussed separately in the paragraphs below.

Phase I surface soil results indicated widespread, elevated concentrations of metals and PAHs in surface soil (0-6 inches [in.] below ground surface [bgs]) throughout the upland area of Iona Island (DU-1 through DU-7, DU-9 through DU-16, and DU-22) (Figure I-2). The highest concentrations of CERCLA hazardous metals in Phase I upland surface soil were detected at DU-4 (copper and zinc), DU-7 (lead), and DU-11 (arsenic). In addition, the highest concentrations of total high molecular weight (HMW) and low molecular weight (LMW) PAHs[2] were detected at DU-13. Contaminant migration pathways to the Hudson River and Iona Marsh shorelines were not identified within the upland area. TAL metals and PAHs in upland soil are unlikely to be transported to the shoreline and be present in shoreline media at concentrations that may pose risk to human health or aquatic ecological receptors. However, metals and PAH concentrations in upland soil may pose risk to terrestrial ecological receptors (plants and soil invertebrates). Further investigation of upland soil is needed to evaluate bioavailability of metals and PAHs to ecological receptors.

Elevated concentrations of metals were reported in hydric surface soil (0–6 in. bgs) and subsurface soil (6–36 in. bgs) at DU-17, DU-18, and DU-19, established within the marsh area between Iona and Round Islands to evaluate area of concern (AOC) #16 – Area D (former Dumping Area) (Figure I-2). Concentrations of metals may pose risk to terrestrial ecological receptors (plants, soil invertebrates, and insectivorous birds and mammals). The highest concentrations of CERCLA hazardous metals in Phase I hydric soil were detected at DU-17 and DU-18, while the highest concentrations of PAHs in hydric soil were detected in surface soil at DU-19. Further investigation of hydric soil is needed to evaluate bioavailability of TAL metals to ecological receptors.

1.1.2 DU-17 through DU-19 (AOC #16 – Area D [Former Dumping Area]): Contaminant Migration Pathways and Shoreline Exposure Routes

Contaminant migration routes to the shoreline were identified for AOC #16 – Area D (former Dumping Area), including tidal flow to the Hudson River shoreline and defined surface water channels leading to Iona Marsh (riprap filled drainage swale and tidal wetland swale) (Figure I-2). Further investigation is needed to evaluate contaminant migration pathways to the Hudson River and Iona Marsh shorelines and evaluate whether metals from AOC #16 – Area D (former Dumping

[2] Ecological risk assessments evaluate PAHs based on their molecular weight and associated toxicity. There are only toxicity reference values for HMW and LMW as total; therefore, in the ERA, PAHs are separated into HMW and LMW groups.

Area) are present in shoreline media at concentrations that may pose risk to human health and/or aquatic ecological receptors.

1.2 HTRW RI PHASE II INVESTIGATION

The HTRW RI Phase II investigation (Phase II) is being conducted to further evaluate migration pathways and exposure routes to support development of the human health risk assessment (HHRA) and ecological risk assessment (ERA).

Phase II includes the following field activities:

- Collection of Incremental sampling methodology (ISM) soil samples from eight Phase I soil DUs, including 5 DUs in the upland area (upland soil) (DU-4, DU-7, DU-9, DU-11, and DU-13) and three DUs in the marsh area between Round and Iona Islands (hydric soil) (DU-17, DU-18, and DU-19) for analysis of physical parameter data (grain size, total organic carbon [TOC], oxidation-reduction potential [ORP], and cation-exchange capacity [CEC]) to assess bioavailability of metals and PAHs across Iona Island and within each soil environment and support development of the ERA (Figure I-3).

- Collection of discrete surface water samples within AOC #16 – Area D (former Dumping Area) (Figure I-3) to evaluate whether contaminants are being mobilized from soil within the source area to tidal water.

- Collection of discrete surface water samples from the Hudson River along the eastern boundary of AOC #16 – Area D (former Dumping Area) to evaluate the potential for migration of metals from AOC #16 – Area D (former Dumping Area) to the river shoreline (Figure I-3).

- Collection of discrete surface water samples along the Iona Marsh shoreline to evaluate potential migration of contaminants from AOC #16 – Area D (former Dumping Area). Sampling locations will be concentrated at the outlets of the riprap filled drainage swale and tidal wetland swale identified during Phase I to further evaluate these swales as contaminant migration pathways.

- Collection of ISM sediment samples from Phase II sediment DUs established along the Hudson River shoreline at the eastern boundary of AOC #16 – Area D (former Dumping Area) and from Iona Marsh at the outlets of the riprap filled drainage swale and tidal wetland swale to evaluate whether metals from AOC #16 – Area D (former Dumping Area) are present in sediment at concentrations that may pose risk to human health and/or ecological receptors (Figure I-3). The ISM sediment samples will also be analyzed for physical parameters (grain size, TOC, pH, and ORP) to support evaluation of bioavailability of metals in each DU and support development of the ERA (Figure I-3).

- Collection of discrete shoreline sediment samples from the Hudson River and Iona Marsh shorelines for analysis of acid volatile sulfide (AVS)/simultaneously extracted metals (SEM) to assess the potential for toxicity to sediment-dwelling organisms and support development of the ERA (Figures I-3 and I-4).

- Collection of discrete surface water and ISM sediment background samples at Con Hook (upstream island within the Hudson River with similar depositional environments to Iona Island) to evaluate whether metals concentrations detected in surface water and sediment at Iona Island are a result of former Department of Defense (DoD) activities or whether concentrations are consistent with background concentrations (Figure I-4).

Laboratory analysis for samples collected during Phase II include the following:

- TAL Metals by U.S. Environmental Protection Agency (EPA) Methods 6010C/6020A
- Mercury by EPA Methods 7470A (water samples) and 7471B (soil samples)
- Hardness by Standard Method (SM) 2340B
- AVS/SEM by SM 4500-S2D and EPA Method 6010C[3]
- Grain size by ASTM International (ASTM) D422[4]
- TOC by Walkley-Black
- CEC by EPA SW-846 Method 9081.

Laboratory analytical services for metals (including mercury), AVS/SEM, grain size, and TOC analyses for will be provided by DoD-certified laboratories as follows. Laboratory certifications are provided in Appendix C.

- Eurofins TestAmerica Canton, OH: ISM sediment preparation
- Eurofins TestAmerica, Denver, CO: metals analyses (including mercury and hardness)
- Eurofins-Lancaster, PA: AVS/SEM analysis
- Eurofins TestAmerica, Corpus Christi, TX: CEC analysis
- Katahdin Analytical Services, LLC, Scarborough, ME: TOC and grain size analyses

ISM sample preparation and laboratory analysis of CEC will be conducted by laboratories that are not DoD-accredited but have been deemed acceptable by the USACE project chemist. Laboratories that are not DoD-accredited will follow QA/QC requirements per the DoD Quality Systems Manual (QSM), Version 5.3 (DoD 2019) to the extent possible.
Field screening will be completed to determine surface water quality parameters (i.e., pH, temperature, specific conductivity, dissolved oxygen [DO], ORP, turbidity, total suspended solids [TSS], and salinity), sediment pH, sediment ORP, and soil ORP.

[3] AVS/SEM involves acidification of the sediment to release the AVS. Sulfide from the AVS is analyzed using SM 4500-S2D. SEM liberated during the acidification process are analyzed via EPA Method 6010C.
[4] The most recent version of ASTM-D422 was withdrawn in 2016 because it was not updated within the required period of time. ASTM D422 has not yet been replaced, and DoD still accredits labs for ASTM D422. ASTM D422 was used to analyze grain size during Phase I and will be used during Phase II for consistency.

This UFP-QAPP Addendum contains modifications and additions to selected worksheets found in the UFP-QAPP (EA 2020a). This UFP-QAPP Addendum, in conjunction with the UFP-QAPP (EA 2020a), documents the following for Phase II:

- Specific investigative procedures and data collection activities
- Laboratory analytical and reporting procedures
- Quality assurance (QA), and quality control (QC) measures and assessments that will be used to confirm that data are of known quality sufficient to meet project objectives for Phase II.

The purpose of this UFP-QAPP Addendum is to provide instruction and guidance associated with Phase II sampling and data analysis to ensure collected data are scientifically valid, meet the established QC objectives, are legally defensible, and support project objectives.

This UFP-QAPP Addendum, in conjunction with the UFP-QAPP (EA 2020a), functions as the Work Plan for EA to implement Phase II to close data gaps identified following completion of Phase I. A summary of the changes to the UFP-QAPP worksheets and appendices is provided below and is followed by the revised versions of the worksheets and appendices on subsequent pages. Remaining worksheets are included in the UFP-QAPP (EA 2020a) in their original format with their original content.

1.3 UFP-QAPP ADDENDUM ORGANIZATION

1.3.1 UFP-QAPP Addendum Worksheet #1 And #2 – Title and Approval Page

Worksheet #1 and #2 presents the title and approval page for this UFP-QAPP Addendum.

1.3.2 UFP-QAPP Addendum Worksheet #4, #7, and #8 – Personnel Responsibilities, Qualifications, and Sign-Off Sheet

Worksheet #4, #7, and #8 has been updated to reflect updates to the EA project personnel.

1.3.3 UFP-QAPP Addendum Worksheet #6 – Communication Pathways

Worksheet #6 has been updated to reflect updates to USACE, EA, and laboratory project personnel.

1.3.4 UFP-QAPP Addendum Worksheet #9 – Project Planning Summary Session

Worksheet #10 has been updated to present information from Technical Project Planning (TPP) Meetings #2 and #3.

1.3.5 UFP-QAPP Addendum Worksheet #10 – Conceptual Site Model

Worksheet #10 has been updated to present the conceptual site model (CSM) for the areas that will be sampled as part of the Phase II investigation.

1.3.6 UFP-QAPP Addendum Worksheet #11 – Project Quality Objectives

Worksheet #11 has been updated to present the data quality objectives (DQOs) for Phase II.

1.3.7 UFP-QAPP Addendum Worksheet #12 – Measurement Performance Criteria Tables

Worksheet #12 in this Addendum presents measurement performance criteria for analysis of TAL metals in water samples by EPA methods 6010C/6020A (Eurofins TestAmerica Denver), mercury in water samples by 7470A (Eurofins TestAmerica Denver), sediment AVS/SEM Eurofins Lancaster Laboratories, Inc.), and soil CEC (Eurofins TestAmerica Corpus Christi).

Eurofins TestAmerica Denver measurement performance criteria tables for TAL metals in soil (and sediment) by EPA Methods 6010C/6020A, mercury in soil by 7471B, are presented in the UFP-QAPP (EA 2020a). Hardness by 2340B is a calculation method and there are no applicable measurement performance criteria tables for hardness. The 6010C metals tables for water presented in this UFP-QAPP Addendum are applicable to the hardness calculation results.

Katahdin Analytical Services, LLC measurement performance criteria tables for TOC by Walkley-Black are presented in the UFP-QAPP (EA 2020a). There are no measurement performance criteria tables for grain size by ASTM D422.

1.3.8 UFP-QAPP Addendum Worksheet #14 and #16 – Project Tasks and Schedule

Worksheet #14 and #16 has been updated to present the Phase II sampling tasks and updated HTRW project schedule.

1.3.9 UFP-QAPP Addendum Worksheet #15 – Reference Limits and Evaluation Tables

Worksheet #15 of presents project action limits (PALs) (i.e., EPA screening values) and achievable laboratory limits for metals (water and sediment samples), AVS/SEM (sediment samples), and CEC (soil samples).

PALs and achievable laboratory limits for TOC analysis in soil and sediment by Walkley-Black are presented in the UFP-QAPP (EA 2020a). There are no PALs or laboratory limits for grain size by ASTM D422.

1.3.10 UFP-QAPP Addendum Worksheet #17 – Sampling Design and Rationale

Worksheet #17 has been updated to present the overall process for the design and rationale of the Phase II sampling program.

1.3.11 UFP-QAPP Addendum Worksheet #18 – Sampling Locations and Methods

Worksheet #18 has been updated to present the location and analytical methods for each sample that is planned to be collected during Phase II.

1.3.12 UFP-QAPP Addendum Worksheet #19 and #30 – Sample Containers, Preservation, and Hold Times

Worksheet #19 and #30 has been updated to present sample containers, preservation requirements, and hold times for each media being sampled and each analysis being conducted during Phase II.

1.3.13 UFP-QAPP Addendum Worksheet #20 Field Quality Control

Worksheet #20 has been updated to present field QC samples for each media being sampled and each analysis being conducted during Phase II.

1.3.14 UFP-QAPP Addendum Worksheet #21 Field Standard Operating Procedures

Worksheet #21 has been updated to present each field Standard Operating Procedure (SOP) that will be used during Phase II. Field SOPs that were not included in the UFP-QAPP (EA 2020a) or that have been updated since finalization of the UFP-QAPP in January 2020 are listed in Worksheet #21 and included in Appendix D of this UFP-QAPP Addendum. These SOPs include EA-SOP-057 ISM, EA-SOP-007 Surface Water Sampling, and EA-SOP-043 Multi-Probe Water Quality Monitoring Instruments.

1.3.15 UFP-QAPP Addendum Worksheet #23 – Analytical Standard Operating Procedure References Table

Worksheet #23 has been updated to present each laboratory SOP that will be used during Phase II. Laboratory SOPs included in Appendix E of this UFP-QAPP Addendum.

1.3.16 UFP-QAPP Addendum Worksheet #24 – Analytical Instrument Calibration Table

Worksheet #24 has been updated to present analytical instrument calibration information for analysis of AVS/SEM (Eurofins Lancaster Laboratories, Inc.) and CEC (Eurofins TestAmerica Corpus Christi).

Eurofins TestAmerica Denver analytical instrument calibration information for TAL metals by EPA Methods 6010C/6020A and mercury by 7471B (soil samples) are presented in the UFP-

QAPP (EA 2020a). Analytical instrument calibration information for mercury by 7470A (water samples) are the same as for mercury by 7471B (soil samples) presented in the UFP-QAPP (EA 2020a). Hardness by 2340B is a calculation method. The 6010C metals information presented in Worksheet #24 of the UFP-QAPP (EA 2020a) is applicable to the hardness calculation.

Katahdin Analytical Services, LLC analytical instrument calibration information for TOC by Walkley-Black is presented in the UFP-QAPP (EA 2020a). There is no calibration information for grain size.

1.3.17 UFP-QAPP Addendum Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Worksheet #25 has been updated to present instrument and equipment maintenance, testing, and inspection information for analysis of AVS/SEM (Eurofins Lancaster Laboratories, Inc.) and CEC (Eurofins TestAmerica Corpus Christi).

Eurofins TestAmerica Denver analytical instrument and equipment maintenance, testing, and inspection information for TAL metals by EPA Methods 6010C/6020A and mercury by 7471B (soil samples) are presented in the UFP-QAPP (EA 2020a). Analytical instrument and equipment maintenance, testing, and inspection information for mercury by 7470A (water samples) are the same as for mercury by 7471B (soil samples) presented in the UFP-QAPP (EA 2020a). Hardness by 2340B is a calculation method. The 6010C metals information presented in Worksheet #25 of the UFP-QAPP (EA 2020a) is applicable to the hardness calculation.

Katahdin Analytical Services, LLC analytical instrument and equipment maintenance, testing, and inspection information for TOC by Walkley-Black, and grain size by ASTM D422 are presented in the UFP-QAPP (EA 2020a).

1.3.18 UFP-QAPP Addendum Worksheet #26 – Sample Handling, Custody, and Disposal

Worksheet #26 has been updated to present Phase II sample nomenclature information.

1.3.19 UFP-QAPP Addendum Worksheet #28 – Quality Control Samples Table

Worksheet #28 has been updated to present corrective action information for analysis of TAL metals in water samples by EPA methods 6010C/6020A (Eurofins TestAmerica Denver), mercury in water samples by 7470A (Eurofins TestAmerica Denver), AVS/SEM (Eurofins Lancaster Laboratories, Inc.) and CEC (Eurofins TestAmerica Corpus Christi).

Eurofins TestAmerica Denver QC and corrective action information for TAL metals in soil (and sediment) samples by EPA Methods 6010C/6020A and mercury in soil (and sediment) samples by 7471B are presented in the UFP-QAPP (EA 2020a).

Hardness by 2340B is a calculation method. The QC and corrective action information for 6010C metals presented in the UFP-QAPP (EA 2020a) is applicable to the hardness calculation results.

Katahdin Analytical Services, LLC measurement performance criteria tables for TOC by Walkley-Black are presented in the UFP-QAPP (EA 2020a). There are no measurement performance criteria tables for grain size.

1.3.20 UFP-QAPP Addendum Worksheet #36 – Data Validation Procedures

Worksheet #36 has been updated to present validation procedures for Phase II analytical data.

1.3.21 UFP-QAPP Addendum Appendix A – Phase I HTRW Technical Memorandum

The Phase I HTRW Technical Memorandum included in Appendix A at the end of this UFP-QAPP Addendum details Phase I field activities and presents Phase I analytical results tables.

1.3.22 UFP-QAPP Addendum Appendix B – Iona Island Shoreline Reconnaissance Survey Technical Memorandum

The Iona Island Shoreline Reconnaissance Survey Technical Memorandum included in Appendix B at the end of this UFP-QAPP Addendum presents the Phase I shoreline reconnaissance field activities and observations.

1.3.23 UFP-QAPP Addendum Appendix C – Laboratory Certifications

Laboratory certifications pertinent to each Phase II analysis are included in Appendix C at the end of this UFP-QAPP Addendum:

- Eurofins TestAmerica Denver DoD (Environmental Laboratory Accreditation Program) ELAP certification
- Eurofins-Lancaster DoD ELAP Certification
- Katahdin Analytical Services, LLC DoD ELAP Certification.

1.3.24 UFP-QAPP Addendum Appendix D – Field Standard Operating Procedures

The following Phase I Field SOPs are applicable to the Phase II. These Field SOPs are presented in Appendix H of the UFP-QAPP (EA 2020a).

- EA-SOP-001: SOP for Sample Labels
- EA-SOP-002: SOP for Chain-of-Custody Form
- EA-SOP-004: SOP for Sample Packing and Shipping
- EA-SOP-005: SOP for Field Decontamination

- EA-SOP-015: SOP for Document Control System, December 2014
- EA-SOP-021: SOP for Sediment Sampling
- EA-SOP-059: SOP for Field Logbook.

Additional Field SOPs pertinent to Phase II that were not included in the UFP-QAPP (EA 2020a) are as follows. These Field SOPs are included in Appendix D at the end of this UFP-QAPP Addendum.

- EA-SOP-007: SOP for Surface Water Sampling
- EA-SOP-043: SOP for Multi-Probe Water Quality Monitoring Instruments
- EA-SOP-057: SOP for ISM.

1.3.25 UFP-QAPP Addendum Appendix E – Laboratory Standard Operating Procedures

Laboratory SOPs pertinent to each Phase II analysis are included in Appendix E at the end of this UFP-QAPP Addendum:

- ISM Sample Preparation (Eurofins TestAmerica Canton):
  - NC-OP-044 Rev 4: Soil Processing Method ASTM D6323-12, Interstate Technology Regulatory Council (ITRC) Guidance Document, Soil Fractionation Method for Michigan
  - NC-OP-046 Rev. 3: Subsampling, ASTM D6323-12
  - WI-NC-0238_030521: Soil Processing – Ball Mill Procedure.
- Sample Analysis (Eurofins TestAmerica Denver):
  - DV-IP-0010 Rev 13: Acid Digestion of Aqueous Samples for Metals Analysis by Inductively coupled plasma (ICP)
  - DV-IP-0014 Rev 12: Acid digestion of Aqueous Samples for Analysis by Inductively coupled plasma mass spectrometry (ICPMS) (SW-846 3005A, 3020A, and EPA 200.8)
  - DV-IP-0015, Rev 14: Acid Digestion of Solids (EPA 3050B)
  - DV-MT-0016 Rev 14: Mercury in Solids by Cold Vapor Atomic Absorption (CVAA) (SW-846 7471AB)
  - DV-MT-0017 Rev 14: Mercury in Solids by CVAA (SW-7470A)
  - DV-MT-0018 Rev 11: ICPMS for Trace Element Analysis by SW-846 Method 6020

— V-MT-0021 Rev 9: ICP Analysis for Trace Elements by SW-846 Method 6010C-D

— DV-OP-0013 Rev 13: ISM for Soils and Sediments (ASTM D 6323)

— DV-OP-0015: Microwave Extraction of Solid Samples by Method [SW-846 3546].

- Sample Analysis AVS (Eurofins Lancaster Laboratories, Inc.):

— TWCWI11483: Colorimetric Sulfide in Waters, Sulfide as Hydrogen Sulfide (H2S), Dissolved Sulfide in Waters by 4500S2 D2011, 4500S2 H2011, or EPA 376.2

— TWCWI10422: AVS in Solids

— TMETWI7920: Dilute/Run and AVS/SEM Sample Handling for Metals

— TMETWI11931: Metals by ICP for Methods SW846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)

- Sample Analysis CEC (Eurofins TestAmerica Corpus Christi):

— CC-ATM-M020: CEC of Soils SW-846 Method 9081 and LDNR Method 29-B

— CC-ATM-M005: Trace Metals Analysis by ICP-Atomic Emission Spectrometry (AES).

- Sample Analysis (Katahdin Analytical Services, LLC):

— CA-551-03: Grain Size Analysis

— CA-777-00: Titrimetric Determination of Organic Carbon Using the Walkley-Black Method

— SD-902-14: Sample Receipt and Internal Control, 05/20, Revision 14

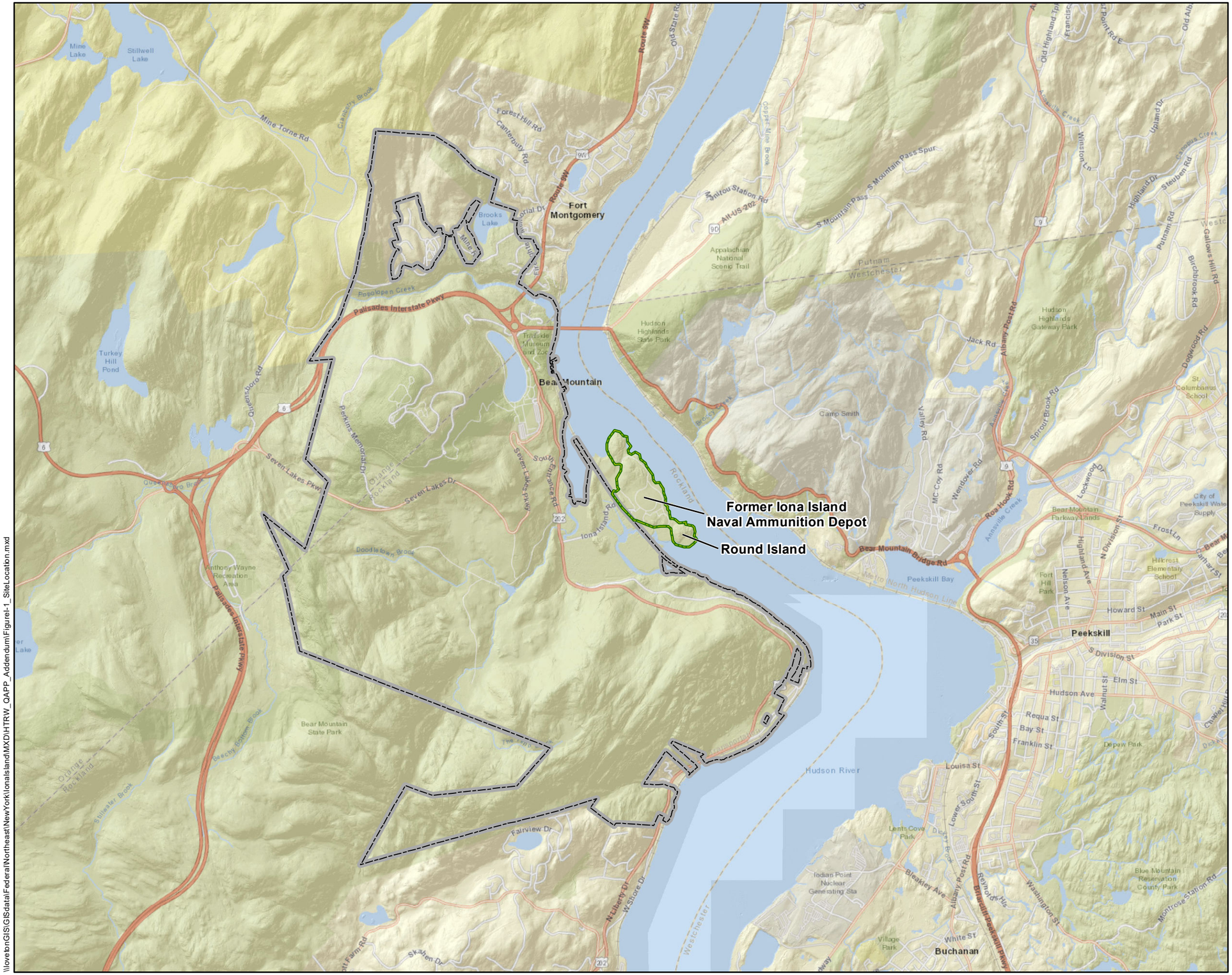
— SD-903-06: Sample Disposal, 09/17, Revision 6.

1.3.26 UFP-QAPP Addendum Appendix F – Field Form

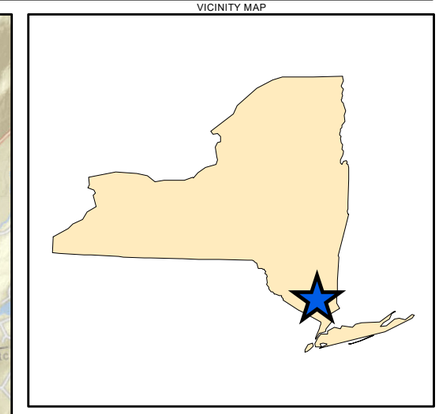
- Water quality meter calibration form.

1.3.27 UFP-QAPP Addendum Appendix G – Supplemental Ecological Risk Assessment Information

Appendix G at the end of this UFP-QAPP Addendum presents sediment and surface water benchmarks, ecological receptor exposure parameters, bioaccumulation factors, and biota-sediment accumulation factors for the ERA.



VICINITY MAP



Legend

- FUDS Boundary
- Bear Mountain State Park Boundary (NYS Office of Parks, Recreation & Historic Preservation, 2018)

0 0.5 1
Miles
1 in = 1 miles

N

FIGURE I-1
General Location of the Iona Island Naval Ammunition Depot Formerly Used Defense Site

HAZARDOUS TOXIC AND RADIOACTIVE WASTE QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Basemap: ESRI World Street Map - ArcGIS Online Map Service (ESRI 2009a).
DEM: 2012, Photo Science, Inc., Coastal New York LiDAR (Hydro Flattened Raster DEM), 1-Meter Resolution, New York State GIS

Map Date: 12/22/2020

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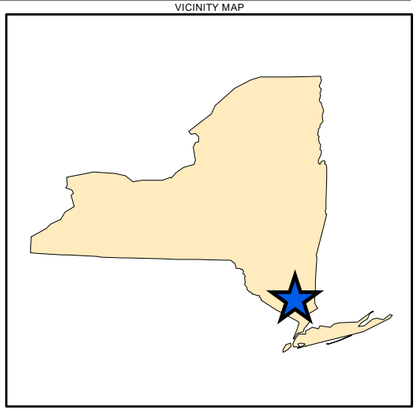
VICINITY MAP



Legend

- FUDS Boundary
- Phase I Former Building/Structure
- Remaining Structure (Not FUDS Eligible)
- Previous Elevated Concentration of COPC (Greeley-Polhemus 1997 and Alion 2008)
- Phase 1 Soil Decision Unit
- Surface Water Flow Direction

0 300 600
Feet
1 in = 300 ft

N

FIGURE I-2
HTRW Areas of Concern and Phase I Decision Units

HAZARDOUS TOXIC AND RADIOACTIVE WASTE QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial: EA Engineering, Science, and Technology. April, 7 2020
AOC Boundary: USAGC 2018

Map Date: 1/31/2022

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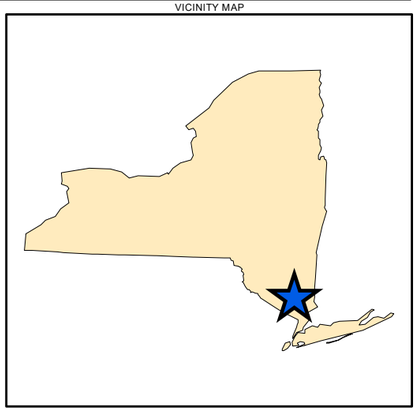
VICINITY MAP



Legend

- FUDS Boundary
- Phase I Former Building/Structure
- AOC #16 – Area D (former Dumping Area)
- Phase I Soil Decision Unit
- Upland
- Marsh/Wetland
- Surface Water Migration Pathway
- Phase II Sediment Decision Unit
- Surface Water Flow Direction

0 200 400
Feet
1 in = 220 ft

N

FIGURE I-3
Phase II Onsite Investigation Areas

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial: EA Engineering, Science, and Technology. April, 7 2020
AOC Boundary: USAGC 2018
Upland and Marsh/Wetland: Cornell Cornell Institute
for Resource Information Sciences (IRIS) 2011

Map Date: 1/31/2022

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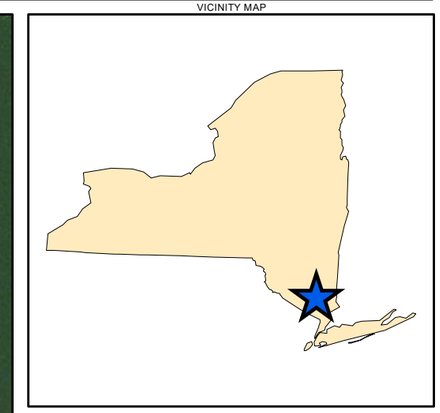
VICINITY MAP



Legend

- FUDS Boundary
- Phase II Background Sediment Sampling Unit
- Surface Water Flow Direction

FIGURE I-4
Phase II Background Investigation Area

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial: ESRI ArcGIS Online Map Service (ESRI 2009a and b).
Inset Basemap: ESRI World Street Map - ArcGIS Online Map Service (ESRI 2009a).
Inset DEM: 2012, Photo Science, Inc., Coastal New York LiDAR (Hydro Flattened Raster DEM), 1-Meter Resolution, New York State GIS

Map Date: 1/31/2022

\\loveton\GIS\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\HTRW_QAPP_Addendum\Figure1-4_PhaseIIBackgroundSamplingArea.mxd

Table 1-1 Summary of Changes associated with the UFP-QAPP Addendum

QAPP Addendum Worksheets	Summary of Changes
Worksheets #1 and #2 - Title and Approval Page and QAPP Identifying Information	Updated title and approval
Worksheets #3 and #5 - Project Organization and QAPP Distribution	No changes
Worksheets #4, #7, #8 - Personnel Qualifications and Sign-off Sheet	Updates to the EA project personnel.
Worksheet #6 - Communication Pathways	Updates to the USACE, EA, and laboratory project personnel
Worksheet #9 - Project Planning Session Summary	Updated to include summaries of Technical Project Planning (TPP) Meetings #2 and #3
Worksheet #10 - Conceptual Site Model	Updates to the conceptual site model (CSM) CSM for the areas that will be sampled as part of the Phase II investigation.
Worksheet #11 - Project/ Data Quality Objectives	Updated to reflect the data quality objectives (DQOs) for Phase II
Worksheet #12 - Measurement Performance Criteria	Updated to reflect the measurement performance criteria for analysis TAL metals in water samples by EPA methods 6010C/6020A (Eurofins TestAmerica Denver), mercury in water samples by 7470A (Eurofins TestAmerica Denver), sediment AVS/SEM (Eurofins Lancaster), and soil CEC (Eurofins TestAmerica Corpus Christi).
Worksheet #13 - Secondary Data Uses and Limitations	No changes
Worksheets #14 and #16 - Project Tasks and Schedule	Updated to present the Phase II sampling tasks and project schedule
Worksheet #15 - Screening Limits and Laboratory- Specific Detection/ Quantitation Limits	Updated to present project action limits (PALs) (i.e., EPA screening values) and achievable laboratory limits for metals (water and sediment samples), AVS/SEM (sediment samples), and CEC (soil samples).
Worksheet #17 - Sampling Design and Rationale	Updated to present the overall process for the design and rationale of the Phase II sampling program.
Worksheet #18 - Sampling Locations and Methods	Updated to present the location and analytical methods for each sample that is planned to be collected during Phase II.
Worksheets #19 and #30 - Sample Containers, Preservation and Hold Times	Updated to present sample containers, preservation requirements, and hold times for each media being sampled and each analysis being conducted during Phase II.
Worksheet #20 - Field Quality Control Summary	Updated to present field QC samples for each media being sampled and each analysis being conducted during Phase II.
Worksheet #21 - Field Standard Operating Procedures	Updated to present each field Standard Operating Procedure (SOP) that will be used during Phase II. Field SOPs that were not included in the UFP-QAPP (EA 2020a) or that have been updated since finalization of the UFP-QAPP in January 2020 are listed in Worksheet #21 and included in Appendix D of this UFP-QAPP Addendum. These SOPs include EA-SOP-057 Incremental Sampling Methodology, EA-SOP-007 Surface Water Sampling, and EA-SOP-043 Multi-Probe Water Quality Monitoring Instruments.
Worksheet #22 - Field Equipment Calibration, Maintenance, Testing and Inspection	No changes
Worksheet #23 - Analytical Standard Operating Procedures	Updated to present each laboratory SOP that will be used during Phase II.
Worksheet #24 - Analytical Instrument Calibration	Updated to present analytical instrument calibration information for analysis of AVS/SEM (Eurofins Lancaster) and CEC (Eurofins-TestAmerica Corpus Christi).
Worksheet #25 - Analytical Instrument and Equipment Maintenance, Testing and Inspection	Updated to present instrument and equipment maintenance, testing, and inspection information for analysis of AVS/SEM (Eurofins Lancaster) and CEC (Eurofins-TestAmerica Corpus Christi).
Worksheets #26 and #27 - Sample Handling, Custody and Disposal	Updated to present Phase II sample nomenclature information.
Worksheet #28 - Analytical Quality Control and Corrective Actions	Updated to present corrective action information for analysis of TAL metals in water samples by EPA methods 6010C/6020A (Eurofins TestAmerica Denver), mercury in water samples by 7470A (Eurofins-TestAmerica Denver), AVS/SEM (Eurofins Lancaster) and CEC (Eurofins-TestAmerica Corpus Christi).
Worksheet #29 - Project Documents and Records	No changes
Worksheets #31, #32 and #33 - Assessments and Corrective Action	No changes
Worksheet #34 - Data Verification and Validation Input	No changes
Worksheet #35 - Data Verification Procedures	No changes

QAPP Addendum Worksheets	Summary of Changes
Worksheet #36 - Data Validation Procedures	Updated to present validation procedures for Phase II analytical data.
Worksheet #37 - Data Usability Assessment	No changes

UFP-QAPP ADDENDUM WORKSHEET #1 AND #2– TITLE AND APPROVAL PAGE

Site Location	Iona Island Naval Ammunition Depot FUDS, Stony Point, Rockland County, New York	
Contract/Work Assignment	W912DR-15-D-0014, Delivery Order #W912DR18F0587	
Contract Title	Iona Island FUDS Military Munitions Response Program (MMRP) and IRP*– Remedial Investigation through Decision Document	
Document Title	UFP-QAPP Addendum No. 1, HTRW Remedial Investigation, Iona Island Naval Ammunition Depot FUDS, Stony Point, Rockland County, New York	
Lead Organization Project Manager (PM)	Erin Kirby, Professional Geologist (P.G.), Licensed Environmental Professional (LEP) USACE–New England District	
Signature:	*Field Copy to be Signed*	**Date:**
Technical Manager (TM)	Todd Beckwith USACE–Baltimore District	
Signature:	*Field Copy to be Signed*	**Date:**
Investigative Organization PM	Michael McGuire EA Engineering, Science, and Technology, Inc., PBC	
Signature:	*Field Copy to be Signed*	**Date:**
Regulatory Agency Regional PM Printed Name/Title Signature/Date	Steven Scharf New York State Department of Environmental Conservation (NYSDEC)	
Signature:	*Field Copy to be Signed*	**Date:**
Stakeholder PM Printed Name/Title Signature/Date	Edwin McGowan, Ph.D. Palisades Interstate Park Commission (PIPC)	
Signature:	*Field Copy to be Signed*	**Date:**
*Installation Restoration Program (IRP) has been changed to HTRW throughout the document per the Project Delivery Team request.		

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UFP-QAPP ADDENDUM WORKSHEET #3 AND #5 – PROJECT ORGANIZATION AND QUALITY ASSURANCE PLAN DISTRIBUTION LIST

This worksheet identifies key project personnel, as well as lines-of-authority and lines-of-communication among the lead organization, prime contractor, subcontractors, and regulatory agencies. This UFP-QAPP Addendum and any changes/revisions will be provided to recipients shown on Table 3-1 below. Contractors and subcontractors shown on this chart are responsible for document control within their organizations.

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Figure 3-1.
Project Organizational Chart

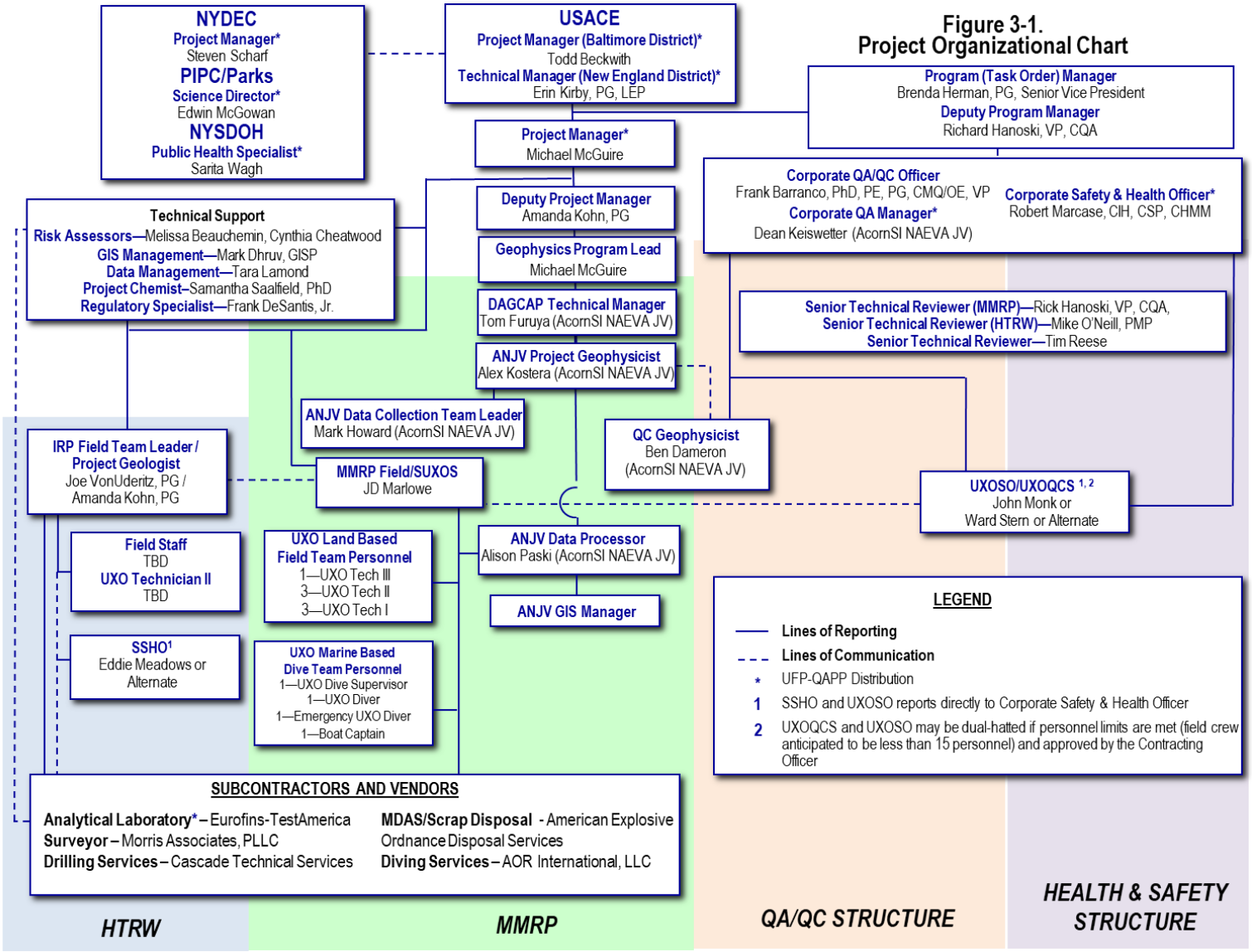

Table 3-1 Responsible UFP-QAPP Recipients

Name	Title	Organization	Telephone Number	Email Address
Erin Kirby	PM	USACE – New England District	978-318-8147	erin.kirby@usace.army.mil
Todd Beckwith	TM	USACE – Baltimore District	410-962-6784	todd.t.beckwith@usace.army.mil
Steven Scharf	PM	NYSDEC	518-402-9620	steven.scharf@dec.ny.gov
Edwin McGowan	Science Director	PIPC/Parks	845-786-2701	edwin.mcgowan@parks.ny.gov
Sarita Wagh	Public Health Specialist	NYSDOH	518 402-7860	Sarita.Wagh@health.ny.gov
Michael McGuire	PM	EA	410-329-5198	mmcguire@eaest.com
Robert (Rob) Marcase	Corporate S&H Manager	EA	410-392-5192	rmarcase@eaest.com

UFP-QAPP ADDENDUM WORKSHEET #4, #7, AND #8 – PERSONNEL RESPONSIBILITIES, QUALIFICATIONS, AND SIGN-OFF SHEET

This worksheet identifies key project personnel for each organization performing tasks defined in this UFP-QAPP Addendum and summarizes their title or role, qualifications (e.g., training and experience), any specialized training, licenses, certifications, or clearances required by the project. The qualifications of USACE personnel are under the purview of the DoD and will not be outlined in this UFP-QAPP Addendum. In addition, federal and state stakeholders' qualifications are under the purview of their respective agencies and will not be presented in this UFP-QAPP Addendum. The table in this worksheet summarizes the responsibilities and provides a space for the signatures of key personnel to the site covered in this UFP-QAPP Addendum. Signatures on the sign-off sheet indicate personnel have read, and agree to implement this UFP-QAPP Addendum, as written. Certification and training records for identified personnel are maintained by the organization's human resources department and are available on request.

Organization: Contractor - EA

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
Brenda Herman, Professional Geologist (P.G.)	Program Manager	**Undergraduate Course Work** • Bachelor of Science (B.S.); Biology; University of Delaware; 1984 **Graduate Course Work** • Master of Science (M.S.); Geology; University of Delaware; 1989 Ms. Herman is a P.G. with 32 years of experience managing and conducting environmental projects for the Department of Defense (DoD). Ms. Herman has been the Program Manager for several Indefinite Delivery/Indefinite Quantity contracts with USACE–Baltimore, Norfolk, and Omaha districts, as well as Aberdeen Proving Ground and Fort Belvoir. These contracts have covered hazardous, toxic, and radiological waste and Military Munitions Response Program (MMRP) investigation and removals; natural resources; compliance; and technology services.	• Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Responses (HAZWOPER) • USACE Construction Quality Management for Contractors • EA Project Manager (PM) Training • Wharton Class, Essentials of Management	• P.G.—Tennessee (No. 4671)	*Field Copy to be Signed*
Richard Hanoski	Deputy Program Manager	**Undergraduate Course Work** • B.S.; Psychology; University of La Vern; 1986 **Graduate Degree** • Master of Public Administration/Management Information Systems/1994 (Troy State University) Mr. Hanoski is a Certified Quality Auditor and Master Explosives Ordnance Disposal (EOD) Technician D with 43 years of experience and is responsible for managing the national Munitions Response Services. He reviews and provides the technical direction for all MRS, including performance and technical oversight of all munitions response site operations and associated activities (digital geophysics, geographic information systems (GIS), logistics, explosives management. He is responsible for all aspects of the MRS and Environmental Remediation divisions nationally and internationally. Performs MRS administrative and operational functions of EA.	• OSHA 40-Hour Training • OSHA 8-Hour Hazardous Waste Operations Supervisor Training • OSHA 8-Hour HAZWOPER Refresher • EA Project Management Training • USACE/Naval Facilities Engineering Command Quality Control (QC) Certification	• Certified Quality Auditor, American Society for Quality • Master EOD Technician	*Field Copy to be Signed*
Michael McGuire	PM, Geophysics Program Lead	**Undergraduate Course Work** • B.S.; Geophysical Engineering; Colorado School of Mines; 1981 Mr. McGuire serves as a Senior Geophysicist in the Munitions Response Group and provides technical support on MMRP projects. He has more than 34 years of experience as a geophysicist including project management, data collection, processing, interpretation, and mapping of seismic reflection and refraction data, time and frequency domain electromagnetic data, magnetic data, ground penetrating radar data, resistivity data, and borehole geophysics. He has extensive experience related to the detection and mapping of unexploded ordnance.	• OSHA 40-Hour HAZWOPER Training; 1989 • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 8-Hour Health and Safety Supervisor Training; Current • Amoco 720-Hour Geologic and Geophysical Training; 1987 • Project Management, Supervision, Public Speaking, Department of Transportation; Tetra Tech, Inc. • ESTCP/NAOC UX-Analyze Advanced Classification Course • U.S. Army Engineering Support Center Huntsville Oasis Montaj UX-Analyze • Pacific Northwest National Laboratories Visual Sample Plan for Munitions and explosives of concern (MEC) Investigations Course • Cardiopulmonary resuscitation (CPR) and First Aid Training; Biannually		*Field Copy to be Signed*

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
Amanda Kohn, P.G.	Deputy PM	**Undergraduate Course Work** • B.S., State University of New York at Cortland; Geology; 2004 Ms. Kohn is a P.G. with 14 years of experience in EA's Site Characterization and Remediation Group. She is experienced in the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process from the Preliminary Assessment/Site Investigation through Remedial Action stage. She has served as the PM for U.S. Environmental Protection Agency (EPA) Superfund Sites and New York State Department of Environmental Conservation (NYSDEC) Superfund Sites. In addition, she has served as the Deputy PM, Task Manager, and Field Team Lead for multiple projects under DoD, EPA, and NYSDEC contracts. Her responsibilities have included tracking and managing budgets and schedules, serving as a point-of-contact with clients, preparing planning documents, coordinating field investigations, overseeing project execution, completing data analysis, and developing and reviewing technical reports.	• OSHA 40-Hour HAZWOPER Training; 2007 • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 8-Hour Health and Safety Supervisor Training; Current • CPR and First Aid Training; Biannually • Project Management Training; 2017 • Senior Technical Review (STR) Training; 2021	• P.G.–Pennsylvania (No. 005105) • P.G.–New York (No. 000115)	*Field Copy to be Signed*
Timothy Reese, Professional Engineer (P.E.)	STR	**Undergraduate Course Work** • B.S; Civil Engineering; Syracuse University; 1989 Mr. Reese has 31 years of PM experience, including over 20 years as PM at munitions sites and has managed MMRP and HTRW projects at 100+ DoD facilities nationwide for the U.S. Army, Air Force, and EPA. He has performed and managed complex environmental, construction and MEC projects. He has successfully completed environmental and munitions projects under various federal regulations including: MMRP, CERCLA, Superfund Amendments and Reauthorization Act (SARA), National contingency Plan (NCP), and Toxic Substances Control (TSCA), as well as guidance provided by the DoD, U.S. Army, USACE, Navy, Air Force, EPA, and state regulators (including the NYSDEC). Over the past 10 years, he has managed 25 MEC task orders at the field operation level at 80+ current and former installations and Formerly Used Defense Sites (FUDS) under MMRP. Base Realignment and Closure, and Installation Restoration Program (IRP) including five Remedial Investigation/Feasibility Studies (RI/FS) involving digital geophysical mapping, munitions constituent sampling, surface/subsurface MEC investigations, MEC and munitions debris clearance, and removal actions.	• OSHA 40-Hour HAZWOPER Training; 1989 • OSHA 8-Hour HAZWOPER Refresher; annually • OSHA 8-Hour Hazardous Waste Operations Supervisor Training; 1990 • Construction Quality Management for Contractors, USACE; 2016 • Geosynthetic Research Institute – Designing with Geosynthetics for Landfill Design, Drexel University • Hazardous Waste/Material Operations Training Course • U.S. Department of Transportation Regulation Training • IATA Training in the Shipment of Dangerous Goods • Visual Sample Plan Training • CPR and First Aid Training; Biannually	• P.E.—New Jersey (No. 39951)	*Field Copy to be Signed*
Cynthia Cheatwood	Human Health Risk Assessor	**Undergraduate Course Work** • B.S.; Civil Engineering; University of Maryland, 1993 **Graduate Course Work** • M.S.P.H.; Environmental Health; Johns Hopkins Bloomberg School of Public Health; 2016 Ms. Cheatwood is an environmental engineer with 28 years of experience who specializes in human health risk assessment (HHRA) and environmental site assessments. As the primary risk assessor for numerous projects, Ms. Cheatwood is familiar with standard HHRA practice and has successfully completed over 200 risk assessments under various regulatory frameworks, including federal, state, and local agencies. She has performed and overseen all aspects of a risk assessment, from determining appropriate sample plans, maintaining databases of chemical data, to using exposure models.	• OSHA, 40-hour HAZWOPER Training, 1993 • OSHA, 8-hour HAZWOPER Supervisor Training, 1996 • Project Management Training; 2007 • CPR and First Aid Training; Biannually		*Field Copy to be Signed*
Melissa Beauchemin	Ecological Risk Assessor	**Undergraduate Degree** • B.S.; Biology; Stonehill College, Easton, MA; 1998 **Graduate Degree** • M.S.; Environmental Science; University of Massachusetts; 2002 Ms. Beauchemin has over 18 years of experience as an environmental scientist, specializing in risk assessments, sediment quality evaluations, remedial investigations, and site-closure documents. She prepares technical documents according to state and federal guidance for complex sites, ranging in size from small local upland habitats to broad-scale offshore assessments, and from the local to national scale. Ms. Beauchemin evaluates potential risks to aquatic and terrestrial organisms from exposure to industrial chemicals in environmental media. She has evaluated risks from persistent environmental chemicals such as polychlorinated biphenyls, dioxins/furans, polycyclic aromatic hydrocarbons (PAHs), pesticides, explosives, and metals (e.g., arsenic, mercury, and lead).	• OSHA, 40-hour HAZWOPER Training, 2005 • OSHA, 8-hour HAZWOPER Refresher, Annually • Project Management Training • STR Training • CPR and First Aid Training; Biannually		*Field Copy to be Signed*

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
Samantha Saalfield, Ph.D.	Project Chemist	**Undergraduate Course Work** • Bachelor of Arts (B.A.); Geology/Chemistry; Whitman College; 2004 **Graduate Course Work** • Ph.D.; Earth Sciences; Dartmouth College; 2009 Dr. Saalfield has 16 years of experience in site characterization and remediation, including inorganic geochemistry, contaminant fate and transport, groundwater modeling, geochemical analysis, and data quality planning and analysis. She specializes in the behavior of inorganic contaminants in surface and groundwater. She also has experience with data quality planning and analysis, assessments of remedial alternatives, and geochemical and hydrogeological modeling.	• OSHA 40-Hour HAZWOPER Training; 2009 • OSHA 8-Hour HAZWOPER Refresher; Annually • Confined Space Training; 2014 • INNOV-X Systems Radiation Safety and Operations Training for Field X-ray fluorescence analyzers • Interstate Technology and Regulatory Council's Incremental Sampling Methodology Training • DoD Quality Systems Manual v.5 Training • EA PM Training • CPR and First Aid Training; Biannually		*Field Copy to be Signed*
Tara Lamond	Data Manager	**Undergraduate Course Work** • B.S.; Environmental Studies; Shepherd University; 2006 Ms. Lamond is a Scientist with 14 years of experience. Her current and past responsibilities have included data management, data evaluation, data visualization, laboratory coordination, soil classification, geotechnical investigations, GIS, environmental site assessments, Uniform Federal Policy (UFP) Quality Assurance Project Plan (QAPP) creation, Sampling and Analysis Plans, project management, and deputy technical chief for environmental data management. She has successfully managed data for several different types of environmental projects ranging from DoD sites, Superfund sites, emergency response (spill) sites, operational ranges, and voluntary cleanup sites. She has experience working with database platforms such as EQuIS, Microsoft Access and Excel, Monitoring and Remediation Optimization System, Environmental Restoration Information System (ERIS), Automated Data Review (ADR), Environmental Resources Program Information Management System (ERPIMS), SCRIBE Environmental Data Management, Environmental Data Management System (EDMS) and the formerly used defense sites online chemical database (FUDSChem).	• OSHA 40-Hour HAZWOPER Training; 2019 • OSHA 8-Hour HAZWOPER Refresher; Annually • Project Management Training • Environmental Restoration Information Systems Training, U.S. Army Environmental Command; 2009 • Environmental Resources Planning Tools (ERPTOOLSX) Training, Air Force Center for Engineering and the Environment; 2011 • EA Supervisor Training; 2016 • CPR and First Aid Training; Biannually		*Field Copy to be Signed*
Frank DeSantis, Jr.	Regulatory Specialist	**Undergraduate Course Work** • B.S.; Environmental and Forest Biology; State University of New York College of Environmental Science and Forestry; 2000 Mr. DeSantis has over 14 years of experience in the biological and environmental fields, including Phase I and Phase II assessments in support of the Army Operational Range Assessment Program, remedial action–operation and long-term management activities in support of the U.S. Air Force and Army Environmental Command environmental programs; site characterizations and pre-design investigations in support of U.S. Air Force performance-based remediation programs; and RIs and FSs and remedial design for the NYSDEC. He has provided project management for preliminary site assessments, site characterizations, RI/FS, and remedial design/remedial actions. He served as the investigation manager for an RI/FS of a metals-impacted tidal marsh at Camp Smith, New York. He understands the New York guidance (e.g., Division of Environmental Remediation [DER]-10: Technical Guidance for SI and Remediation) and promulgated remedial regulations (e.g., Title 6, New York Codes Rules and Regulations, Part 375, Soil Clean-up Objectives) at sites with metal contaminants showing migration via surface water and sediment deposition.	• OSHA 40-Hour HAZWOPER Training • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 8-Hour Hazardous Waste Operations Supervisor Training • 30-hour Construction Safety and Health • Permit Required Confined Space Training • Safe-Capture: Chemical Immobilization of Wildlife • CPR and First Aid Training; Biannually		*Field Copy to be Signed*
Joe Von Uderitz, P.G.	Project Geologist	**Undergraduate Course Work** • B.S.; Environmental Studies and Geology; Alfred University; 2001 Mr. Von Uderitz is a P.G. with more than 12 years of experience in environmental site assessment and remediation. His primary responsibilities are oversight of field activities including preparing work plans, scheduling, and overseeing implementation of work plans. Oversight includes Contractor QC responsibilities of the installation and operations of treatment systems, as well as installation of monitoring and recovery wells, and development, gauging, and sampling of wells. He maintains product recovery, dual phase extraction systems, and catalytic oxidizer systems at various locations. Additionally, he has experience with construction activities and associated safety and health concerns, site inspections to evaluate work conditions for potential job site hazards, ensuring proper selection and use of personal protective equipment, use of monitoring equipment, and safety and health compliance. Provides daily health and safety briefings for personnel on multiple construction sites. Provides technical review and assisted in preparation of safety procedures for Site Safety and Health Plans and APPs.	• OSHA 40-Hour HAZWOPER Training • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 30-hour Construction Safety and Health Training • USACE Construction Quality Management for Contractors Worker • The Mine Safety and Health Administration Training • 40-hour Radiation Worker I Training • Excavation Safety Training (Trenching/Shoring) • Association of Reciprocal Safety Councils Training • Permit-Required Confined Space Entrant • Permit-Required Confined Space Attendant • Permit-Required Confined Space Supervisor • General Radiation Worker Training • CPR and First Aid Certified	• P.G.- Pennsylvania (No. 005197) • P.G.-New York (No. 000144)	*Field Copy to be Signed*

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
Amanda Kohn, P.G.	Field Team Lead / Project Geologist	Undergraduate Course Work • B.S., State University of New York at Cortland; Geology; 2004 Ms. Kohn is a P.G. with 14 years of experience in EA's Site Characterization and Remediation Group. She is experienced in the CERCLA process from the PA/SI through Remedial Action stage. She has worked on numerous site characterization, RI, qualitative and quantitative site assessment, and remediation projects involving free-phase and dissolved-phase petroleum hydrocarbon plumes, chlorinated solvent plumes, and metals-contaminated sites for numerous state and federal projects, as well as DoD facilities. She has completed multiple analytical sampling projects and served as the team leader, task manager, and geologist for numerous sampling events. Her field experience includes subcontractor oversight during drilling and excavation activities, field screening and classification of soil and bedrock, sampling of various environmental media, collection of field data, and monitoring of water quality parameters.	• OSHA 40-Hour HAZWOPER Training; 2007 • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 8-Hour Health and Safety Supervisor Training; Current • CPR and First Aid Training; Biannually • Project Management Training; 2017 • STR Training; 2021	• P.G.–Pennsylvania (No. 005105) • P.G.–New York (No. 000115)	*Field Copy to be Signed*
Michael O'Neill, PMP	STR	**Undergraduate Course Work** • B.S.; Engineering Science; Loyola College; 1990 Mr. O'Neill is a PM with 31 years of experience in every facet of the CERCLA process. He has 23 years of experience successfully completing environmental projects under Federal regulations (CERCLA, Resource Conservation and Recovery Act [RCRA], SARA, TSCA, and MMRP) as well as DoD and U.S. Army guidance. He has been involved in many phases of site assessments (Phase I and Phase II site assessments, environmental baseline surveys and engineering FSs), and RIs beginning with development of the scope of work and preparation of the field sampling plan, preparation of health and safety plans, site and subcontractor management, site research, sampling and sample custody, as well as report preparation, remedial design, project costing and oversight during remedial action. He has experience managing various size tasks and projects.	• OSHA 40-Hour HAZWOPER Training • OSHA 8-Hour Hazardous Waste Operations Supervisor Training • Asbestos Inspector • Lead Inspector (EPA Agency Model Curriculum) • MARSSIM Radiological Survey Design Course • Introductory Health Risk Communications Workshop • Construction Quality Management for Contractors • EA Project Management Training • Unexploded ordnance (UXO) Safety Training Program • Anti-Terrorism Level I Training • CPR and First Aid Training; Biannually	• Project Management Institute (No. 1273776)	*Field Copy to be Signed*
Frank Barranco, Ph.D., P.G., P.E.	Corporate QC Manager	**Undergraduate Course Work** • B.A., Geology; Duke University; 1984 **Graduate Course Work** • M.S.; Geology; University of Texas Arlington; 1988 • Ph.D., Environmental Science and Engineering; Colorado School of Mines; 1998 Dr. Barranco has 31 years of experience in the development and implementation of Quality Assurance (QA)/QC systems. As the Director of QC for EA's Quality Management Program, Dr. Barranco authors, and implements company-wide QA and QC policies, guidance documents, and SOPs. In this role, Dr. Barranco is heavily involved in the quality monitoring associated with QC systems across EA, including the STR Program, Project and Program Reviews, and oversight of the Technical Chiefs. In addition to this company-wide role, Dr. Barranco serves as QA Officer on individual programs and large Indefinite Delivery/Indefinite Quantity contracts across EA.	• OSHA 40-Hour HAZWOPER Training • OSHA 8-Hour Hazardous Waste Operations Supervisor Training • CPR and First Aid Training; Biannually	• P.G.–Tennessee (No. 005603) • P.E. –Maryland (No. 43120) • P.G.-New York (No. 001202-1) • Certified Manager of Quality/ Operational Excellence (No. 53424)	*Field Copy to be Signed*
Robert (Rob) Marcase, Certified Industrial Hygienist (CIH), Certified Safety Professional (CSP), Certified Hazardous Materials Manager (CHMM)	CIH	**Undergraduate Course Work** • B.S. Environmental Science (Minor in Geology), Slippery Rock University; 1995 Mr. Marcase is a CIH, CSP, and CHMM with 25 years of experience in a wide range of environmental, safety and health, and industrial hygiene projects. His experience includes regulatory compliance strategy, industrial hygiene investigation and process analysis, occupational exposure assessment, facility-wide health and safety auditing, and air quality permitting and compliance. His experience also includes environmental assessments, building inspections, industrial air pollution testing, and water quality analysis.	• OSHA 40-Hour HAZWOPER Training • OSHA30-Hour Construction Safety Training • National Institute for Occupational Safety and Health 582 Certification, Collecting and Analyzing Asbestos Air Samples; 1998 • Asbestos Building Inspector Certification, Maryland and Pennsylvania, 1999	• CIH—2007 (No. 9283CP) • CSP—2010 (No. 21609) • CHMM–2012 (No. 15935)	*Field Copy to be Signed*

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
John "JD" Marlowe	Senior Unexploded Ordnance Supervisor (SUXOS)	**Education** • Graduate of Naval EOD School; Indian Head, Maryland; 1995 Mr. Marlowe has 42 years of experience in all phases of munitions response actions and applicable safety standards, including supporting DoD installation operations and IRP remedial investigations. He has extensive EOD/ UXO experience in all phases of munitions response actions, including SUXOS experience on MMRP action projects for the DoD. He is experienced in performing and supervising MEC projects and has extensive "hands-on" training to include all aspects of safety; extensive project and technical management, field operations and crew leadership; and expertise in MEC/UXO identification and recovery. Has 18 years of QA/QC experience in supervisory EOD/UXO positions overseeing project health and safety matters, including oversight and interface for onsite subcontractors; implementing safety meetings, visitor orientations, inspections, training, audits, and self-assessments; providing site-specific MEC awareness training; coordination with range control and adherence to installation procedures; and maintaining written safety and equipment logs/records on DoD project across the United States. Mr. Marlowe is experienced in complying with CERCLA/RCRA; DoD; EPA Region 3, and NCP regulations. His munitions experience includes, projects related to improvised explosive devices, small arms ammunition, general purpose bombs, cluster bombs, pyrotechnics, artillery projectiles, rockets, missiles, and incendiary and chemical bombs.	• OSHA 40-Hour HAZWOPER; 1996 • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 8-Hour Hazardous Waste Operations Supervisor; 2014 • OSHA 30-Hour Construction; 2013 • OSHA 10-Hour Construction; 2017 • Naval School, Explosive Ordnance Disposal Phase 2 (surface); 1995 • Hazardous Material Safety (Canadian); 1994 • Transportation of Dangerous Goods (Canadian); 1994 • EOD/Improvised Explosive Device Course (Canadian); 1993 • DEODS NATO Officers/SNCO EOD (England) Course; 1992 • EOD Specialty Course (Canadian); 1988 • EOD Basic Course (Canadian); 1979 • OSHA Annual Respirator Medical; 2018 • Forestry-Granton Institute of Technology; 1992 • Electronics-Saskatchewan Technical Institute; 1983 • Level 1 Anti-Terrorism Training • NAVSEA Material Potentially Presenting an Explosive Hazard (MPPEH) Training, 2018 • CPR and First Aid Training; Biannually	• Master EOD Technician • HFA Certified Heavy Equipment Operator • Range Safety Officer Certification (Canadian)	*Field Copy to be Signed*
John Monk	UXO Quality Specialist (QCS) and UXO Safety Officer (UXOSO)	**Education** • Naval EOD Mr. Monk is a Senior UXO Technical Expert with over 36 years of experience in Naval EOD/diver and munitions and MEC projects. His primary responsibilities are as a UXOQCS/UXOSO and team leader for munitions response related projects. His project work focuses primarily on location, removal/disposal, and clearance of conventional, chemical/biological/radiological munitions. He is also an expert in underwater ordnance and Naval underwater diving techniques for both nitrogen and mixed gas diving operations. He has experience with identifying and resolving safety and quality issues with respect to munitions related activities. He functions as a liaison with USACE and subcontractors' safety personnel and quality personnel, and experience with oversight of all aspects of explosive safety on various DoD projects.	• EA PM Training; 2008 • OSHA 40-hour HAZWOPER Training; 1994 • OSHA 8-Hour Site Managers and Supervisors Training Course; 2015 • OSHA 8-Hour HAZWOPER Refresher; Annually • OSHA 30-Hour Construction Safety Course; 2005 • OSHA 8-Hour Hazardous Waste Operations Supervisor Training • Hazardous Waste Management and Shipping for Environmental Professionals; 2001 • USACE Construction Quality Management for Contractors Course; 2017 • Delaware Department of Natural Resources and Environmental Control Soil and Water Conservation (Blue Card) Course; 2005 • Competent Person Trenching and Excavation, West Virginia University; 2002 • Fall Prevention and Protection Competent Person Training; 2002 • Confined Space Course Certificate; 2011 • Master EOD Technician; 1996 • Explosives Blasters License-MD #G-300 • CPR and First Aid Training; Biannually		*Field Copy to be Signed*

Name	Project Title/Role	Education/Experience	Specialized Training	Licenses/ Certifications	Signature/Date
Eddie Meadows	Site Health and Safety Officer	**Undergraduate Course Work** • B.S.; Environmental Sciences; Virginia Tech; 1995 Mr. Meadows has 22-year of experience in the successful completion of numerous projects and field efforts or phases of those projects, which include management; field oversight; health and safety officer; sample collection and sample custody of soil, water, sediment, and air matrixes; mobile laboratory testing; field screening; report writing, and data reduction/management; basic surveying; and remedial injection activities. He has served as Site Safety and Health Officer for several investigations, removal actions, groundwater monitoring well installations, and construction projects.	• OSHA 40-Hour HAZWOPER • OSHA 8-Hour Hazardous Waste Operations Supervisory Training • OSHA 30-Hour Construction Safety and Health Certification • Excavation Safety for Competent Person Training • Confined Space Training • USACE Construction Quality Management for Contractors • OSHA 24-Hour Fall Protection for Competent Person Training • Immunoassay Training for field screening of Polycyclic Aromatic Hydrocarbons, Polychlorinated Biphenyls, and Trinitrotoluene • X-Ray Fluorescence Spectrum Analyzer Training • Basic Radiation Worker and Fundamentals of Radiation Training. • Respirator Fit Test		*Field Copy to be Signed*

Organization: Laboratory – Eurofins TestAmerica

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Dylan Bieniulis	PM	On file with laboratory	On file with laboratory	*Field Copy to be Signed*
Roxanne Sullivan	QA Supervisor	On file with laboratory	On file with laboratory	*Field Copy to be Signed*
Richard Clinkscales	Laboratory Director	On file with laboratory	On file with laboratory	*Field Copy to be Signed*

Organization: Laboratory – Eurofins Lancaster Laboratories

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Robert Dempsey	Eurofins Lancaster Lab President	On file with laboratory	On file with laboratory	*Field Copy to be Signed*
Dorothy Love	Eurofins Lancaster QA Director	On file with laboratory	On file with laboratory	*Field Copy to be Signed*

Organization: Laboratory – Katahdin Analytical Services, LLC (To be Added Upon Receipt from Laboratory)

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
TBD	TBD	On file with laboratory	On file with laboratory	*Field Copy to be Signed*

UFP-QAPP ADDENDUM WORKSHEET #6 – COMMUNICATION PATHWAYS

This worksheet documents specific issues (communication drivers) that will trigger the need for formal (documented) communication with other project personnel or stakeholders. Its purpose is to ensure there are procedures in place for providing notifications, obtaining approvals, and generating the appropriate documentation when handling important communications, including those involving regulatory interfaces, approvals to proceed from one Definable Feature of Work to the next, field changes, emergencies, non-conformances, and stop-work orders.

Communication Drivers	Responsible Entity	Name	Telephone Number	Procedure (Timing, Pathways, etc.)
Contractual modification and/or program performance	USACE Contracting Officer	Kathryn Brown (Baltimore District)	410-962-2585	Overall responsibility for overseeing and monitoring the contractor's performance and assures the contractor receives impartial, fair, and equitable treatment under the Delivery Order. Ultimately responsible for the final determination of the adequacy of the contractor's performance and authorized to obligate the Government on this Delivery Order.
Contractual modification and/or program performance	USACE Contract Officer Representative	Sesh Lal (Baltimore District)	410-962-2585	Contractual modification and/or program performance. Communicates directly with USACE PM, USACE TM, and EA PM/Deputy PM.
PM with USACE	USACE PM	Erin Kirby, P.G., LEP	978-318-8147	Primary USACE POC. Overall management of the project. Communicates with and gives direction to EA PM. Reviews and approves project plans and their modifications. Communicates with regulators and relays information to EA PM/Deputy PM.
TM with USACE	USACE TM	Todd Beckwith	410-962-6784	Communicates with USACE PM and gives direction to EA PM. Reviews and approves project plans and their modifications.
POC with NYSDEC	NYSDEC	Steven M. Scharf, P.E.	845-786-2701	Case Manager and primary POC for State of New York. Provides regulatory oversight. Communicates with USACE PM and/or PIPC Executive Director.
POC with PIPC	PIPC Director of Science	Edwin McGowan, Ph.D.	845-786-2701 ext. 263	Primary POC for PIPC. Provides stakeholder oversight. Communicates with USACE PM and/or NYSDEC.
Contractual modification and/or program performance	EA Program Manager	Brenda Herman, P.G.	410-527-2474	Communication with USACE at the Programmatic Level regarding overall performance. Acts as an advocate for USACE to ensure that project needs are met. Supports the EA PM/Deputy PM. Provides corporate commitment to allocate necessary resources.
Contractual modification and/or program performance	EA STR, Deputy EA Program Manager	Richard Hanoski	443-632-4887	Communication with USACE at the Programmatic Level regarding overall performance. Acts as an advocate for USACE to ensure that project needs are met. Supports the EA PM/Deputy PM. Provides corporate commitment to allocate necessary resources.
Manage all project phases/overall technical lead	EA PM	Mike McGuire	443-986-2488	Overall contractor management of the project. Maintains lines of communication between USACE and EA. Communicates with USACE POC regarding project direction as well as notification of any project issues. Ensures that project is on time and within budget. Ensures that project team maintains technical, quality, and safety and health standards. Leads communication with stakeholders. Manages subcontractors. Completes final review of all deliverables.
Contractor communication with USACE	EA PM	Mike McGuire	443-986-2488	POC for all technical, QA, and administrative matters regarding the Contractor's implementation of the project (verbal, written or electronic).
Modifications to Contractor Program	EA PM	Mike McGuire	443-986-2488	Coordinate fieldwork, programmatic direction, and issue resolution.
Project coordination and support to EA PM/ Deputy PM	EA STR	Timothy Reese, P.E. Michael O'Neill, PMP	410-329-5198 410-329-5142	STR for technical approach and documents. Communicates with the EA PM/Deputy PM.
Change to UFP-QAPP	EA Risk Assessor	Melissa Beauchemin Cynthia Cheatwood	401-287-0380 410-329-5154	Communicates with the EA PM/Deputy PM. Will notify EA PM of approval of minor change (verbal, written, or electronic); complete field change request for efficiency or changed conditions.
Change to UFP-QAPP	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Communicates with the EA PM/Deputy PM. Will notify EA PM of approval of minor change (verbal, written, or electronic); complete field change request for efficiency or changed conditions.
Change to UFP-QAPP	EA PM, Deputy PM	Mike McGuire Amanda Kohn	443-986-2488 315-565-6548	For field change requests, the QAPP does not need to be revised, a field change request should be sent to the USACE PM and USACE TM for approval. In the case of a QAPP addendum, amendment or revision, the QAPP must go through the USACE and EPA for approval.
Project completion and performance deficiencies	Corporate Quality Manager	Frank Barranco, Ph.D. P.E., P.G., CMQ/OE	410-329-5137	Monitor work, procedures, and documentation to ensure compliance with QC procedures and engineering practices. Develops and oversees implementation of a corrective action plan, if necessary. Reviews all aspects of project completion and stop work if deficiencies in the work are noted. Communicates with the EA PM/Deputy PM.
Mobilization and demobilization	EA PM	Mike McGuire	443-986-2488	Communication with USACE PM and USACE TM regarding timelines and access requirements. USACE PM and USACE TM to relay information to other parties.
Coordination of field activities	EA Field Team Leader	Amanda Kohn	315-565-6548	Coordination of Site activities with sub-contractors and communication of site issues and progress with the EA PM/Deputy PM, USACE PM, and USACE TM.

Communication Drivers	Responsible Entity	Name	Telephone Number	Procedure (Timing, Pathways, etc.)
Modifications to/deviations from UFP-QAPP once in Execution	EA PM	Mike McGuire	443-986-2488	A written field change request will be submitted to the USACE PM for modifications to content within the final approved UFP-QAPP.
Modifications to Technical Direction or Modification of Approved Planning Documents	EA PM	Mike McGuire	443-986-2488	Will direct the field team/ SSHO in the implementation of the field activities.
Coordination of sample bottle/sample receipt and analytical data processing	Eurofins TestAmerica, Eurofins Lancaster Laboratory PM	Dylan Bieniulis	303-736-0138	Coordination of sample bottles, confirmation of samples, issues at the laboratory, and data availability will be communicated to the Project Chemist and/or Data Manager.
Technical support	EA SUXOS	John "JD" Marlowe	443-632-4887	Provides technical support for all munitions related projects. Coordination of field activities and oversight for project team to ensure compliance with documents. Coordination of field activities and oversight for project team to ensure compliance with documents. Manages on-land site operations. Reports to EA PM/Deputy PM regarding issues with cost, schedule, etc. Ensures modifications meet USACE requirements and overall project quality objectives. Ensures compliance with approved project documents during execution of field work. Prepares and submits SUXOS Daily Reports daily during land-based field activities.
Quality deficiencies or inconsistencies with plan execution	EA UXOSO/ QCS	John Monk	717-887-5582 (Monk cell)	Provides technical support for all munitions related projects. Coordination of field activities and oversight for project team to ensure compliance with documents. Coordination of field activities and oversight for project team to ensure compliance with documents. Manages on-land site operations. Reports to EA PM/Deputy PM regarding issues with cost, schedule, etc. Ensures modifications meet USACE requirements and overall project quality objectives. Ensures compliance with approved project documents during execution of field work. Prepares and submits SUXOS Daily Reports daily during land-based field activities.
Laboratory oversight	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Provides oversight of laboratory sample analysis and FUDS Chem activities (event planning, uploading SEDD files by lab, and data validation).
Data oversight	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Provides technical oversight of data evaluation and summary in RI Report.
Analytical Corrective Actions	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Report on the adequacy, status, and effectiveness of the QA program to EA PM/Deputy PM.
Technical issues during implementation of the project	EA PM	Mike McGuire	443-986-2488	Will notify the USACE PM and USACE TM of any significant technical or QC issues during the field investigation.
Modifications to analytical laboratory responsibilities	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Notify EA PM/Deputy PM within one week of performance problems encountered by the analytical laboratories by phone, email, or fax.
Field and analytical corrective actions	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	Communication with EA PM/Deputy PM regarding QA issues.
Modifications to analytical responsibilities	Eurofins TestAmerica, Eurofins Lancaster Laboratory PM	Dylan Bieniulis	303-736-0138	The Regional Sample Control Center Coordinator will report project nonconformance issues within one week to the Project Chemist by phone, email, or fax.
Modifications to analytical QA responsibilities	Eurofins TestAmerica, , Eurofins Lancaster Laboratory QA Manager	Roxanne Sullivan	303-736-0116	Report project nonconformance issues within one week to the Laboratory PM in person or by phone, email, or fax.
Release of data	EA Project Chemist	Samantha Saalfield, Ph.D.	410-584-7000 ext. 5215	No analytical data will be released until review is complete and the Project Chemist has approved the release.
Review of project deliverables	USACE Project Chemist	Constance Lapite	978-318-8452	Review of UFP QAPP and data validation reports.
FUDSChem Correspondence	Synectics	Emily Hill	916.737.4010	Communications with EA Data Manager, Laboratory PM, and USACE FUDSChem POC.
FUDSChem Correspondence	EA Data Manager	Tara Lamond	410-329-5172	Communications with Laboratory PM, Synectics and USACE FUDSChem POC.
FUDSChem Correspondence	USACE FUDSChem POC	Brian Jordan	505-506-2189	Review of FUDSChem
FUDSChem Correspondence	USACE FUDSChem POC	Dion Lewis	978-318-8785	Communications with EA Data Manager, Laboratory PM, and Synectics
UXO Emergency in the field	EA UXOSO/QCS and SUXOS	John Monk/ JD Marlowe/ Ward Stern	Monk - 717-887-5582 (cell) Marlowe - 443-752-1775 (cell) Stern - 256-731-9151	Notify the EA PM/Deputy PM, who will relay emergency to appropriate entities. Provides direction to field staff and subcontractors.

Communication Drivers	Responsible Entity	Name	Telephone Number	Procedure (Timing, Pathways, etc.)
Safety incident, stop work order	EA UXOSO/QCS and SUXOS, EA SSHO	John Monk/ JD Marlowe/	Monk - 717-887-5582 (cell) Marlowe - 443-752-1775 (cell)	Notify the EA PM/Deputy PM who will relay information to appropriate entities. Provides direction to field staff and subcontractors.
Emergency in the field	SSHO	Eddie Meadows	410-527-2425	Notify the EA PM/Deputy PM who will relay emergency to appropriate entities. Provides direction to field staff and subcontractors.
Emergency in the field	EA Certified Industrial Hygienist	Robert (Rob) Marcase, CIH, CSP, CHMM	410-392-5192	Notify the EA PM/Deputy PM, who will relay emergency to appropriate entities. Provides direction to field staff and subcontractors.

NOTES:
EA = EA Engineering, P.C. and Its Affiliate EA Science and Technology
EPA = U.S Environmental Protection Agency
FUDS = Formerly Used Defense Site
FUDSChem = Formerly Used Defense Site Online Chemical Database
NYSDEC = New York State Department of Environmental Conservation
PIPC = Palisades Interstate Park Commission
PM = Project Manager
POC = Point-of-contact
QA = Quality assurance
QC = Quality control
QCS = Quality Control Specialist
QAPP = Quality Assurance Protection Plan
RI = Remedial Investigation
SSHO = Site Safety and Health Officer
STR = Senior technical review
SUXOS = Senior Unexploded Ordnance Supervisor
TM = Technical Manager
UFP = Uniform Federal Policy
USACE = U.S. Army Corps of Engineers
UXO = Unexploded Ordnance
UXOSO = Unexploded Ordnance Safety Officer

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UFP-QAPP ADDENDUM WORKSHEET #9 – PROJECT PLANNING SUMMARY SESSION

Two TPP Meetings were conducted to present Phase I field activities and results to stakeholders and to gain government and stakeholder input and document decision points regarding potential implementation of Phase II data gap collection and analysis. The meetings are summarized below.

9.1 TPP MEETING #2

Title: TPP #2
Meeting Location: Teleconference
Date of Session: 15 July 2020
Scoping Session Purpose: Update stakeholders on field activities conducted to date for the project and path forward.

Participants:

Name of Attendee	Organization	Title
Todd Beckwith	CENAB	Technical Support
David King	CENAB	Geophysicist
Erin Kirby	CENAE	Project Manager
Cheryl Montgomery	ERDC/CENAE	Ecological Risk Assessor
Michael Narcisi	CENAE	Ecological Risk Assessor
Michelle Bourne	ERDC/CENAE	Ecological Risk Assessor
Cynthia Auld	CENAE	Human Health Risk Assessor
Drew Clemmons	CENAE	Geologist
Yixian Zhang	CENAE	Chemist
Steven Scharf	NYSDEC	Project Manager
Edwin McGowan	PIPC/Parks	Science Director
Mike McGuire	EA	Project Manager
Amanda Kohn	EA	Deputy Project Manager
Melissa Beauchemin	EA	Ecological Risk Assessor
Cynthia Cheatwood	EA	Human Health Risk Assessor

Notes:
CENAB = United States Army Corps of Engineers – Baltimore District
CENAE = United States Army Corps of Engineers – New England District
EA = EA Engineering, P.C. and Its Affiliate EA Science and Technology
ERDC = Engineering Research & Development Center
NYSDEC = New York State Department of Environmental Conservation
PIPC = Palisades Interstate Park Commission

9.1.1 Notes/Comments relevant to HTRW

9.1.1.1 Overview of HTRW Project

Nineteen areas of concern (locations/footprints of former buildings/structures) were identified for further investigation based on previous investigations and a review of site history. Contaminants

being evaluated during the RI, based on historical information, include PAHs, metals; explosives; benzene, ethylbenzene, toluene, xylenes (BTEX), and/or polychlorinated biphenyls (PCBs).

The HTRW investigation is being conducted in two phases, with each phase including a field investigation and data evaluation/reporting. Phase I includes onsite soil sampling using ISM, completion of shoreline reconnaissance to supplement the CSM and completion of background soil sampling using ISM to establish regional background metals and PAH concentration ranges. Phase I field activities completed to date include onsite and background incremental sampling methodology (ISM) soil sampling and the shoreline reconnaissance. The Phase I data evaluation is ongoing and includes data validation, comparison of analytical results to screening criteria and background concentrations, and completion of baseline human health and ecological risk assessments.

9.1.1.2 Phase I Soil Sampling

A total of 32 onsite DUs were established to evaluate concentrations in surface soil at 0-6 in. bgs and subsurface soil (at 6 to 36 in. bgs, if present). ISM soil sampling was conducted to evaluate the presence/absence and nature/extent of COPCs. To allow calculation of the 95 percent upper confidence limit (UCL) of the arithmetic mean to evaluate concentrations for each DU, replicate samples were collected at onsite locations, with each replicate consisting of the initial ISM sample plus two additional samples (triplicate samples).

ISM surface soil samples were collected using an ISM sampling tool with a 6-inch core barrel. Surface soil sampling was conducted across approximately 25 acres (30 onsite DUs), with a total of 90 ISM surface soil samples (initial and replicate samples) collected for analysis.

Subsurface soil sampling was attempted at each of the 30 onsite DUs using a hand auger. The majority of soil cover across Iona Island is shallow (<12 inches depth). Exposed and shallow bedrock and hardscape was encountered across the majority of the site. Due to shallow soil and shallow depth to refusal, subsurface soils were only collected from 5 onsite DUs including each DU within the former dump area [DU-17 through DU-19], and two upland DUs (DU-20 and DU-21).

DU-8 (established to evaluate AOC #5 – Former Building 207 [Marine Garage Building] was not sampled due to the presence of numerous rusted and deteriorating 55-gallon drums, deteriorating pallets, piles of roofing shingles, chain link fencing, and other debris. The material contained within the drums is not known. DU-8 was not sampled as it would not be possible to separate metals and PAH contaminants originating from former DoD operations at Building 207 from the contaminants originating within the drums and other debris observed in the DU. PIPC has accepted responsibility for the drums/debris observed at DU-8 and NYSDEC concurs that the PIPC is responsible. One additional DU (DU-24) was not sampled as this DU was located in an area with little/no soil cover.

9.1.1.3 Background Soil Sampling

Background soil sampling was conducted to establish regional PAHs and metals concentrations that could be used to evaluate concentrations on Iona Island (determine if concentrations on the island are similar to or greater than background). Eight non-contiguous DUs were established in Bear Mountain State Park in areas based on mapped soil units on Iona Island. Locations were refined based on conditions encountered in the field (organic soil content / organic cover, soil color, grain size, consistency, type). Single surface soil ISM samples were collected from each DU at 0-6 in. bgs. Subsurface soil was not collected at background DUs as soil was less than 12 inches deep. Depths to refusal in soil borings ranged from 3 to 8 inches.

9.1.1.4 Shoreline Reconnaissance

Shoreline reconnaissance was conducted on 6-7 April 2020 and included characterization of four shoreline areas and the Iona Island upland area, collection of sediment samples for analysis of TOC and grain size to evaluate if sediment could be a sink for contaminants, and characterization of upland and shoreline flora and fauna.

Potential pathways identified from onshore to offshore included outfall pipes along the Hudson River shoreline, a groundwater seep along the Iona Marsh shoreline near the parking area, and a tidal wetland swale and drainage swale along the Iona Marsh shoreline between Round Island and Iona Island.

Macroinvertebrate habitat was documented along the Hudson River shoreline and invertebrates (amphipods and snails) were observed on the underside of large stones. The Iona Marsh shoreline was dominated by cattails and phragmities and contained finer-grained sediments.

A driving reconnaissance upstream of Iona Island was conducted to identify potential background sediment sampling locations. Con Hook and Constitution Island were identified as potential background locations. Mr. McGowen stated that a former superfund site is located north of Constitution Island and the PDT discussed potential sampling impacts related to the superfund site. Con Hook is owned by Scenic Hudson. Ms. Kirby stated that USACE is in the process of obtaining rights of entry (ROEs) for both Con Hook and Constitution Island.

9.2 TPP MEETING #3

Title: TPP #3
Meeting Location: Teleconference
Date of Session: 12 November 2020
Scoping Session Purpose: Update stakeholders on project status, including the results of the Phase I sampling, and discuss path forward.

Participants:

Name of Attendee	Organization	Title
Todd Beckwith	CENAB	Project Manager
Erin Kirby	CENAE	Technical Lead
Michelle Bourne	ERDC/CENAE	Ecological Risk Assessor
Cynthia Auld	CENAE	Human Health Risk Assessor
Drew Clemens	CENAE	Geologist
Yixian Zhang	CENAE	Chemist
Constance Lapite	CENAE	Chemist
Steven Scharf	NYSDEC	Project Manager
Michael Narcisi	CENAE	Ecological Risk Assessor
Edwin McGowan	PIPC/Parks	Science Director
Sarita Wagh	NYSDOH	N/A
John Swartwout	NYSDEC	N/A
Mike McGuire	EA	Project Manager
Amanda Kohn	EA	Deputy Project Manager
Vince Williams	EA	Senior Scientist/Operations Manager
Melissa Beauchemin	EA	Ecological Risk Assessor
Cynthia Cheatwood	EA	Human Health Risk Assessor
Erica Thielman	EA	N/A

Notes:
CENAB = United States Army Corps of Engineers – Baltimore District
CENAE = United States Army Corps of Engineers – New England District
EA = EA Engineering, P.C. and Its Affiliate EA Science and Technology
N/A = Not Applicable
NYSDEC = New York State Department of Environmental Conservation
NYSDOH = New York State Department of Health
PIPC = Palisades Interstate Park Commission

9.2.1 Notes/Comments relevant to HTRW

Notes/comments from TPP #3 that are relevant to the HTRW, excluding content covered in TPP #2 under Section 9.1, are presented in the sections below.

9.2.1.1 Phase I Sediment Sampling

Sediment samples were collected at seven locations for analysis of TOC and grain size to evaluate sediment physical characteristics and whether sediment has potential to accumulate historical contaminants. Samples within Iona marsh were fine grained (silt and clay) with high TOC content, indicating sediment within the marsh are likely to accumulate historical contaminants. Samples along the Hudson River shoreline were coarse grained (gravel and sand) with low TOC content, indicating sediment along the river is less likely to accumulate historical contaminants.

9.2.1.2 Phase I Data Analysis, Validation, and Usability Assessment

ISM samples were processed and analyzed by TestAmerica. Preliminary analytical reports and electronic data deliverables uploaded to FUDSChem (central repository for all FUDS data).

Stage 2B Validation Electronic and Manual (S2BVEM) was completed in FUDSChem. A Stage 2B electronic and manual validation was completed by the EA project chemist and data manager. Validation reports were approved by the USACE chemist in June 2020.

As summarized in the data usability assessment (DUA), 27.5 percent of the dataset was qualified as estimated due to trace detections, detections in laboratory blanks, and data quality issues related to ISM procedures and methods (exceeding holding times during laboratory drying of samples). PAH holding times were exceeded as samples needed to air dry prior to processing times. However, PAHs in samples were high molecular weight, which are not prone to degradation.

9.2.1.3 Phase I Results, Risk Assessments, and Phase II Recommendations

The RI is being conducted in accordance with CERCLA using EPA screening criteria, including EPA residential screening levels (RSLs) for soil and EcoSSLs. Industrial soil RSLs are being considered for comparative purposes only. Metals data are also being compared to background data using permutation test of means to evaluate whether concentrations are consistent with or exceed background concentrations. Based on elevated concentrations above both screening criteria and background concentrations, additional evaluation is needed during Phase II to refine exposure estimates. Phase II is anticipated to include surface water and sediment sampling. Background sampling will also be collected at Con Hook, located upstream of Iona Island, to evaluate background concentrations in Hudson River water and sediment.

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UFP-QAPP ADDENDUM WORKSHEET #10 – CONCEPTUAL SITE MODEL

This worksheet presents updated site information for the Iona Island Naval Ammunition Depot FUDS and the project's CSM based on the results of Phase I.

10.1 HTRW REMEDIAL INVESTIGATION PHASE I INVESTIGATION

Phase I was conducted to evaluate the following:

- Presence/absence and/or nature and extent of contaminants in onsite soil
- Regional background soil concentrations for TAL metals and PAHs
- Source-pathway-receptor interaction
- Potential risk to human and ecological receptors exposed to onsite soil.

A total of 32 onsite DUs (DU-1 through DU-25, EXDU-1 through EXDU-7) and 8 background DUs (BADU-1 through BADU-8, established in Bear Mountain State Park) were established to evaluate contaminants in soil during Phase I (Figure I-2). Phase I field activities were conducted in March and April 2020 in accordance with the UFP-QAPP (EA 2020a). Onsite and background ISM soil sampling field methods and soil analytical results tables are presented in the Final Phase I HTRW Technical Memorandum that was submitted to USACE on 4 September 2020 (presented in Appendix A of this UFP-QAPP Addendum). The results of the shoreline reconnaissance survey are presented in the Final Iona Island Shoreline Reconnaissance Survey Technical Memorandum that was submitted to USACE on 15 April 2020 (presented in Appendix B of this UFP-QAPP Addendum). A summary of Phase I results is presented in the subsections below.

10.1.1 Areas Not Investigated During Phase I

Two onsite DUs, DU-8 and DU-24, were not sampled during Phase I. DU-8 was not sampled due to the presence of debris observed within the DU that was not related to historical DoD operations, including numerous rusted and deteriorating 55-gal drums, deteriorating pallets, piles of roofing shingles, and chain-link fencing. PIPC has accepted responsibility for the drums/debris observed at DU-8 and the NYSDEC concurs that the PIPC is responsible for this area. DU-24 was not sampled as the majority of the DU consists of exposed bedrock with little to no soil cover.

10.1.2 Areas Recommended for No Further Investigation

Eleven of 32 onsite Phase I DUs (DU-20, DU-21, DU-23, DU-25, and EXDU-1 through EXDU-7) are recommended for no further investigation during Phase II as either analytical results were below EPA screening criteria and/or background concentrations or preliminary evaluations of risk did not indicate that concentrations of COPCs posed potential risk to terrestrial ecological receptors (Figure I-2).

10.1.3 Areas Recommended for Further Investigation

Metals and/or PAH concentrations in soil at 16 upland DUs (upland soil) and 3 DUs in the marsh area (hydric soil) between Round and Iona Islands (AOC #16 – Area D [former Dumping Area]) were reported above both EPA screening criteria (EPA residential RSLs and/or EcoSSLs) and background concentrations during Phase I (Figure I-2 and Table 10-1).

- DU-1: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #17 – Area E (Former Coal Trestle and Storage Area) and AOC #10 – Former Building 406 (Tin, Electrical and Annealing Shop Building)

- DU-2: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #17 – Area E (Former Coal Trestle and Storage Area)

- DU-3: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #12 – Former Building 410 (Power House) and AOC #11 – Former Building 407 (Paint Shop and Pipe Shop)

- DU-4: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at previous SI sampling location II-EA-SD-02-01 (Alion 2008) (which had elevated concentrations of antimony, copper, lead, and mercury) and previous Data Collection Activities sampling location IX407-B1 (which had elevated concentrations of arsenic, lead, and fluoranthene) (Greeley-Polhemus 1997)

- DU-5: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at AOC #3 – Former Building 123 (Garage Building) and previous SI sampling location II-EA-SS-02-06 (which had elevated concentrations of antimony, copper, lead, mercury, and zinc) (Alion 2008)

- DU-6: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of previous SI sampling locations II-EA-SS-02-09 (which had an elevated concentration of copper) and EA-SS-02-03 (which had elevated concentration of lead) (Alion 2008)

- DU-7: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of previous SI sampling location II-EA-SS-02-08 (which had elevated concentrations of copper, lead, and zinc) (Alion 2008).

- DU-9: 200 ft x 200 ft DU established in the upland area of Iona Island to evaluate potential contamination in upland soil at and in the vicinity of AOC #8 – Former Building 219 (Garage Building) and AOC #9 – Former Building 220 (Garage Building)

- DU-10: 200 ft x 200 ft DU established to evaluate potential contamination in upland soil of Iona Island at and in the vicinity previous sampling location II-EA-SS-02-14 (which had a reported elevated concentration of copper during the 2008 SI) (Alion 2008)

- DU-11: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #18 – Area F (Former Coal Storage Area)

- DU-12: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #6 – Former Building 213 (Paint Locker for Building 202) and AOC #7 – Former Building 215 (Garage Building)

- DU-13: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of AOC #1 – Former Building 103 (Paint and Oil Storage) and previous Data Collection Activities sampling location IX-103-B1 (which had elevated concentrations of arsenic, cadmium, lead, fluoranthene, and pyrene) (Greeley-Polhemus 1997)

- DU-14: 200 ft x 200 ft upland DU established to evaluate potential contamination in soil at and in the vicinity of previous SI sampling location II-EA-SS-02-13 (which had elevated concentrations of lead and mercury) (Alion 2008)

- DU-15: 200 ft x 200 ft DU established in the upland area of Iona Island to evaluate potential contamination in upland soil at and in the vicinity of previous sampling location II-EA-SS-02-15 (which had reported elevated concentrations of lead and mercury during the 2008 SI) (Alion 2008)

- DU-16: 200 ft x 200 ft DU established in the upland area of Iona Island to evaluate potential contamination in upland soil at and in the vicinity of AOC #4 – Former Buildings 124-128 (Sewage Disposal Plant)

- DU-17: 200 x 200 ft DU established in the marsh area between Round and Iona Islands to evaluate potential contamination in soil at and in the vicinity of AOC #2 – Former Building 121 (Incinerator), AOC #16 – Area D (Dumping Area), and previous sampling locations IX-121-B1 (which had elevated concentrations of arsenic, cadmium, lead, fluoranthene, and pyrene) and II-EA-SD-02-02 (which had elevated concentrations of antimony, copper, lead, nickel, and zinc)

- DU-18: 200 x 200 ft DU established in the marsh area between Round and Iona Islands to evaluate potential contamination in soil at AOC #16 – Area D (Dumping Area) and in the vicinity of previous sampling locations IX DMP-B22 (which had elevated concentrations of arsenic, cadmium, chromium, lead, silver, 2,4-dinitrotoluene [DNT], 2,6-DNT, and Aroclor 1260) and IXDMP-B1 (which had elevated concentrations of lead and Aroclor 1260).

- DU-19: 200 x 200 ft DU established to evaluate potential contamination in soil at AOC #16 – Area D (Dumping Area) and in the vicinity of previous sampling location II-EA-SS-02-16 (which had elevated concentrations of antimony, copper, lead, mercury, and zinc).

- DU-22: 60 ft x 60 ft upland DU established to evaluate potential contamination in soil at two former ASTs located near former Building 407 (AOC #15 – Area C).

10.1.3.1 Upland Surface Soil Metals Concentrations

Preliminary evaluations of risk indicate that select metals in Phase I upland surface soil (0-6 in. bgs) (specifically, arsenic, barium, copper, lead, manganese, and zinc) may pose risk to terrestrial plants and soil invertebrates. Concentrations in upland surface soil are as follows:

- Arsenic: concentrations ranging from 6.5 mg/kg at DU-10 to 43 mg/kg at DU-11, and 95UCL ranging from 7.56 mg/kg at DU-16 to 54.2 at DU-4. Background concentrations for arsenic ranged from 2.1 to 7.8 mg/kg, with a 95UCL of 8.59. The EcoSSL for arsenic in soil is 6.8 mg/kg (EPA 2018). For reference, the EPA residential RSL for arsenic in soil is 0.68 mg/kg (EPA 2021).

- Barium: concentrations ranging from 55 mg/kg at DU-6 to 110 mg/kg at DU-7, and 95UCL ranging from 59.5 mg/kg at DU-10 to 129 mg/kg at DU-7. Background concentrations for barium ranged from 13 to 47 mg/kg, with a 95UCL of 56.7 mg/kg. The EcoSSL for barium in soil is 110 mg/kg (EPA 2018). For reference, the EPA residential RSL for barium in soil is 1,500 mg/kg (EPA 2021).

- Copper: concentrations ranging from 5.6 mg/kg at DU-7 to 600 mg/kg at DU-13, and 95UCL ranging from 7.25 mg/kg at DU-7 to 885 mg/kg at DU-13. Background concentrations for copper ranged from 4.9 to 26 mg/kg, with a 95UCL of 27.7 mg/kg. The EcoSSL for copper in soil is 28 mg/kg (EPA 2018). For reference, the EPA residential RSL for copper in soil is 310 mg/kg (EPA 2021).

- Lead: concentrations ranging from 31 mg/kg at DU-10 to 2,000 mg/kg at DU-7, and 95UCL ranging from 42 mg/kg at DU-10 to 3,080 mg/kg at DU-7. Background concentrations for lead ranged from 33 to 94 mg/kg, with a 95UCL of 82.1 mg/kg. The EcoSSL for lead in soil is 11 mg/kg (EPA 2018). For reference, the EPA residential RSL for lead in soil is 400 mg/kg (EPA 2021).

- Manganese: concentrations ranging from 410 mg/kg at DU-15 to 1,100 mg/kg at DU-13, and 95UCL ranging from 465 mg/kg at DU-15 to 1,320 mg/kg at DU-13. Background concentrations for manganese ranged from 37 to 340 mg/kg, with a 95UCL of 381 mg/kg. The EcoSSL for manganese in soil is 11 mg/kg (EPA 2018). For reference, the EPA residential RSL for manganese in soil is 180 mg/kg (EPA 2021).

- Zinc: concentrations ranging from 55 mg/kg at DU-10 to 280 mg/kg at DU-4, and 95UCL ranging from 64.7 at DU-10 to 374 mg/kg at DU-4. Background concentrations for zinc ranged from 21 to 72 mg/kg, with a 95UCL of 69.1 mg/kg. The EcoSSL for zinc in soil is 46 mg/kg (EPA 2018). For reference, the EPA residential RSL for zinc in soil is 2,300 mg/kg (EPA 2021).

Note that types of contamination that may be eligible for DERP FUDS response include CERCLA hazardous substances or pollutants or contaminants, military munitions, POL, or unsafe conditions posed by buildings or structures. Metals that are CERCLA hazardous substances include arsenic cadmium, chromium, copper, lead, nickel, selenium, and silver. The full metals list is reported for completeness purposes, however metals that are not a CERCLA hazardous substance (for example, manganese) are not eligible for DERP FUDS response.

10.1.3.2 Upland Surface Soil PAH Concentrations

Surface soil samples from 12 upland DUs (DU-01 through DU-05, DU-09, DU-11 through DU-13, DU-16, and DU-20 through DU-22) were sampled for PAHs during Phase I based on previous investigation results and COPCs identified for each AOC. The highest concentrations of PAHs in upland surface soil (0–6 in. bgs) were reported at AOC #1 – Former Building 103 (Paint and Oil Storage) as presented below.

- Benzo(a)anthracene: 9.4 to 17 mg/kg and 95UCL of 24.5 mg/kg. Concentrations in the remaining 11 upland DUs sampled for PHAs during Phase I ranged from 0.13 to 0.86 mg/kg. Background concentrations ranged from 0.043 to 0.68 mg/kg, with a 95UCL of 0.727 mg/kg. The EPA residential RSL and EcoSSL for benzo(a)anthracene in soil is 1.1 mg/kg (EPA 2021 and EPA 2018).

- Benzo(a)pyrene: 7.1 to 13 mg/kg and 95UCL of 18.6 mg/kg. Concentrations in the 11 additional upland DUs sampled for PHAs during Phase I ranged from 0.061 to 2.7 mg/kg. Background concentrations ranged from 0.045 to 0.79 mg/kg, with a 95UCL of 0.783 mg/kg. The EPA residential RSL for benzo(a)pyrene in soil is 0.11 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

- Benzo(b)fluoranthene: 9.7 to 17 mg/kg and 95UCL of 25.2 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.11 to 3.9 mg/kg. Background concentrations ranged from 0.072 to 1.1 mg/kg, with a 95UCL of 1.24 mg/kg. The EPA residential RSL and EcoSSL for benzo(b)fluoranthene in soil is 1.1 mg/kg (EPA 2021 and EPA 2018).

- Benzo(g,h,i)perylene: 4.2 to 7.9 mg/kg and 95UCL of 11.4 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.037 to 1.5 mg/kg. Background concentrations ranged from 0.035 to 0.68 mg/kg, with a 95UCL of 0.632 mg/kg. There is no EPA residential RSL for benzo(g,h,i)perylene in soil (EPA 2021). The EcoSSL is 1.1 mg/kg (EPA 2018).

- Benzo(k)fluoranthene: 3.8 to 7.4 mg/kg and 95UCL of 10.5 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.032 to 1.2 mg/kg. Background concentrations ranged from 0.028 to 0.46 mg/kg, with a 95UCL of 0.451 mg/kg. The EPA residential RSL for benzo(k)fluoranthene in soil is 11 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

- Dibenz(a,h)anthracene: 1.2 to 2.4 mg/kg and 95UCL of 3.64 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.011 to 0.45 mg/kg. Background concentrations ranged from 0.0063 to 0.15 mg/kg, with a 95UCL of 0.161 mg/kg. The EPA residential RSL for dibenz(a,h)anthracene in soil is 0.11 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

- Chrysene: 9.3 to 16 mg/kg and 95UCL of 23.5 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.097 to 3.1 mg/kg. Background concentrations ranged from 0.061 to 1 mg/kg, with a 95UCL of 0.992 mg/kg. The EPA residential RSL for chrysene in soil is 110 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

- Fluoranthene: 21 to 38 mg/kg and 95UCL of 53.4 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.11 to 7.9 mg/kg. Background concentrations ranged from 0.12 to 2.2 mg/kg, with a 95UCL of 2.14 mg/kg. The EPA residential RSL for fluoranthene in soil is 240 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

- Indeno(1,2,3-cd)pyrene: 4.9 to 9.2 mg/kg and 95UCL of 12.9 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.042 to 1.8 mg/kg. Background concentrations ranged from 0.037 to 0.74 mg/kg, with a 95UCL of 0.692 mg/kg. The EPA residential RSL and EcoSSL for indeno(1,2,3-cd)pyrene in soil is 1.1 mg/kg (EPA 2021 and EPA 2018).

- Pyrene: 17 to 27 mg/kg and 95UCL of 41.8 mg/kg. Concentrations in remaining upland DUs sampled for PHAs during Phase I ranged from 0.17 to 5.7 mg/kg. Background concentrations ranged from 0.11 to 1.9 mg/kg, with a 95UCL of 1.79 mg/kg. The EPA residential RSL for pyrene in soil is 180 mg/kg (EPA 2021), and the EcoSSL is 1.1 mg/kg (EPA 2018).

Preliminary evaluations of risk indicate that total concentrations of HMW and LMW PAHs[5] may pose risk to insectivorous birds and mammals, terrestrial plants, and soil invertebrates. Total HMW and LMW PAH concentrations were as follows:

[5] Ecological risk assessments evaluate evaluates PAHs based on their molecular weight and associated toxicity. Toxicity reference values are used to evaluate potential risk to wildlife through food web modeling and are typically treated as sum HMW and sum LMW PAHs.

- Total HMW PAHs: concentrations ranging from 1.3 mg/kg at DU-1 to 191.85 mg/kg at DU-13, and 95UCL ranging from 2.11 mg/kg at DU-1 to 275 mg/kg at DU-13. Background concentrations for total HMW PAHs ranged from 0.55 to 9.9 mg/kg, with a 95UCL of 9.59 mg/kg. The EcoSSL for total HMW PAHs in soil is 1.1 mg/kg (EPA 2018). For reference, there is no EPA residential RSL for total HMW PAHs.

- Total LMW PAHs: concentrations ranging from 0.26 mg/kg at DU-1 to 64 mg/kg at DU-13, and 95UCL ranging from 0.41 mg/kg at DU-1 to 91.7 mg/kg at DU-13. Background concentrations for total LMW PAHs ranged from 0.1 to 1.548 mg/kg, with a 95UCL of 1.46 mg/kg. The EcoSSL for LMW PAHs in soil is 29 mg/kg (EPA 2018). For reference, there is no EPA residential RSL for total LMW PAHs.

Note that select DUs (e.g., DU-01, DU-02, and DU-11) were investigated owing to former coal storage activities, and surface soil PAH detections within DUs associated with former coal storage areas may be owing to coal particles. In addition, onsite has been significantly reworked and removed during demolition of former structures between November 1965 and December 1973 and excavation and regrading following demolition. Contaminants including particles of coal remaining from coal storage areas present in surface and shallow subsurface soil were likely spread over much of the operational area. PAH detections owing to particles of coal are not addressed under CERCLA as coal is considered a product rather than a hazardous waste. No remedial actions are warranted owing to PAH detections from coal particles. PAH detections released from coal particles and absorbed on to (e.g., subsurface) soils would be actionable under CERCLA.

10.1.3.3 Hydric Surface and Subsurface Soil Metals and PAH Concentrations

Hydric surface soil (0-6 in. bgs) and subsurface soil (0.6-36 in. bgs) at AOC #16 – Area D (former Dumping Area) was collected from DU-17, 18, and 19 during a low tide event. Samples were described as moist to very moist (not saturated), and sample depths were above free-standing water as encountered in test pits during the MMRP intrusive investigation (3-4 ft bgs). AOC #16 – Area D (former Dumping Area) is inundated during high tide and storm events.

Preliminary evaluations of risk indicate that select metals in Phase I hydric soil at DU-17 and DU-18 (specifically lead, cadmium, and copper) may pose risk to terrestrial birds and mammals. In addition, concentrations of metals (specifically, antimony, arsenic, barium, cadmium, chromium, cobalt, copper, lead, manganese, mercury, nickel, thallium, and zinc) and HMW PAHs at DU-17, DU-18, and DU-19 may pose risk to terrestrial plants and soil invertebrates. Concentrations in hydric soil are as follows:

- Antimony: concentrations in surface soil (0–6 in. bgs) ranging from 0.28 mg/kg at DU-19 to 9.9 mg/kg at DU-17, and 95UCL ranging from 0.749 mg/kg at DU-19 to 12.4 mg/kg at DU-17. Concentrations in subsurface soil (6-36 in. bgs) ranging from 0.11 mg/kg at DU-19 to 19.5 mg/kg at DU-18, and 95 UCL ranging from 0.128 mg/kg at DU-19 to 19.6 mg/kg at DU-18. Background concentrations for antimony ranged from 0.53 to 1.2 mg/kg, with

a 95UCL of 1.35 mg/kg. The EcoSSL for antimony in soil is 0.27 mg/kg (EPA 2018). For reference, the EPA residential RSL for antimony in soil is 12.4 mg/kg (EPA 2021).

- Arsenic: concentrations in surface soil (0–6 in. bgs) ranging from 2.7 mg/kg at DU-17 to 15 mg/kg at DU-18, and 95UCL ranging from 15 mg/kg at DU-17 to 23.1 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 2.6 mg/kg at DU-19 to 23 mg/kg at DU-17, and 95 UCL ranging from 3.15 mg/kg at DU-19 to 27.2 mg/kg at DU-17. Background concentrations for arsenic ranged from 2.1 to 7.8 mg/kg mg/kg, with a 95UCL of 8.59 mg/kg. The EcoSSL for arsenic in soil is 6.8 mg/kg (EPA 2018). For reference, the EPA residential RSL for arsenic in soil is 0.68 mg/kg (EPA 2021).

- Cadmium: concentrations in surface soil (0–6 in. bgs) ranging from 0.25 mg/kg at DU-19 to 20 mg/kg at DU-17, and 95UCL ranging from 0.395 mg/kg at DU-19 to 33.2 mg/kg at DU-17. Concentrations in subsurface soil (6-36 in. bgs) ranging from 0.087 mg/kg at DU-19 to 24 mg/kg at DU-18, and 95 UCL ranging from 0.105 mg/kg at DU-19 to 38.3 mg/kg at DU-18. Background concentrations for cadmium ranged from 0.11 to 0.29 mg/kg, with a 95UCL of 0.274 mg/kg. The EcoSSL for cadmium in soil is 0.36 mg/kg (EPA 2018). For reference, the EPA residential RSL for cadmium in soil is 7.1mg/kg (EPA 2021).

- Chromium: concentrations in surface soil (0–6 in. bgs) ranging from 6.9 mg/kg at DU-17 to 240 mg/kg at DU-18, and 95UCL ranging from 11.8 mg/kg at DU-19 to 411 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 7.7 mg/kg at DU-19 to 33 mg/kg at DU-18, 95 UCL ranging from 7.91 mg/kg at DU-19 to 36.6 mg/kg at DU-18. Background concentrations for chromium ranged from 3.6 to 31 mg/kg, with a 95UCL of 29.9 mg/kg. The EcoSSL for chromium in soil is 23 mg/kg (EPA 2018). For reference, the EPA residential RSL for chromium in soil is 12,000 mg/kg (EPA 2021).

- Cobalt: concentrations in surface soil (0–6 in. bgs) ranging from 4.9 mg/kg at DU-17 to 19 mg/kg at DU-19, and 95UCL ranging from 8.04 mg/kg at DU-18 to 22.7 mg/kg at DU-19. Concentrations in subsurface soil (6-36 in. bgs) ranging from 5 mg/kg at DU-18 to 18 mg/kg at DU-19, and 95 UCL ranging from 7.2 mg/kg at DU-17 to 19.5 mg/kg at DU-19. Background concentrations for cobalt ranged from 0.59 to 7.3 mg/kg, with a 95UCL of 7.83 mg/kg. The EcoSSL for cobalt in soil is 13 mg/kg (EPA 2018). For reference, the EPA residential RSL for cobalt in soil is 2.3 mg/kg (EPA 2021).

- Copper: concentrations in surface soil (0–6 in. bgs) ranging from 40 mg/kg at DU-18 to 1,700 mg/kg at DU-18 and 95UCL ranging from 217 mg/kg at DU-19 to 3,010 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 130 mg/kg at DU-17 to 5,600 mg/kg at DU-18, and 95 UCL ranging from 210 mg/kg at DU-19 to 6,070 mg/kg at DU-18. Background concentrations for copper ranged from 4.9 to 26 mg/kg, with a 95UCL of 27.7 mg/kg. The EcoSSL for copper in soil is 28 mg/kg (EPA 2018). For reference, the EPA residential RSL for copper in soil is 310 mg/kg (EPA 2021).

- Lead: concentrations in surface soil (0–6 in. bgs) ranging from 38 mg/kg at DU-19 to 2,900 mg/kg at DU-17, and 95UCL ranging from 93.9 mg/kg at DU-19 to 4,150 mg/kg at DU-17. Concentrations in subsurface soil (6-36 in. bgs) ranging from 11 mg/kg at DU-19 to 2,900 mg/kg at DU-18, and 95 UCL ranging from 16.2 mg/kg at DU-19 to 4,070 mg/kg at DU-17. Background concentrations for lead ranged from 33 to 94 mg/kg, with a 95UCL of 82.1 mg/kg. The EcoSSL for lead in soil is 11 mg/kg (EPA 2018). For reference, the EPA residential RSL for lead in soil is 400 mg/kg (EPA 2021).

- Manganese: concentrations in surface soil (0–6 in. bgs) ranging from 380 mg/kg at DU-17 to 550 mg/kg at DU-18, and 95UCL ranging from 484 mg/kg at DU-17 to 652 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 17 mg/kg at DU-17 to 510 mg/kg at DU-18, and 95 UCL ranging from 170 mg/kg at DU-17 to 555 mg/kg at DU-18. Background concentrations for manganese ranged from 37-330 mg/kg, with a 95UCL of 381 mg/kg. The EcoSSL for manganese in soil is 220 mg/kg (EPA 2018). For reference, the EPA residential RSL for manganese in soil is 180 mg/kg (EPA 2021).

- Mercury: concentrations in surface soil (0–6 in. bgs) ranging from 0.11 mg/kg at DU-19 to 8.0 mg/kg at DU-18, and 95UCL ranging from 0.822 mg/kg at DU-19 to 8.4 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 0.12 mg/kg at DU-19 to 8.1 mg/kg at DU-18, and 95 UCL ranging from 0.402 mg/kg at DU-19 to 8 mg/kg at DU-18. Background concentrations for mercury ranged from 0.120 to 0.320 mg/kg, with a 95UCL of 0.351mg/kg. The EcoSSL for mercury in soil is 0.013 mg/kg (EPA 2018). For reference, the EPA residential RSL for mercury in soil is 1.1 mg/kg (EPA 2021).

- Nickel: concentrations in surface soil (0–6 in. bgs) ranging from 5.9 mg/kg at DU-17 to 30 mg/kg at DU-30, and 95UCL ranging from 18.1 mg/kg at DU-19 to 44.7 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 14 mg/kg at DU-19 to 79 mg/kg at DU-18, and 95 UCL ranging from 14 mg/kg at DU-19 to 99 mg/kg at DU-18. Background concentrations for nickel ranged from 2.7 to 23 mg/kg, with a 95UCL of 27.6 mg/kg. The EcoSSL for nickel in soil is 38 mg/kg (EPA 2018). For reference, the EPA residential RSL for nickel in soil is 150 mg/kg (EPA 2021).

- Thallium: concentrations in surface soil (0–6 in. bgs) ranging from 0.10 mg/kg at DU-17 to 0.21 mg/kg at DU-18, and 95UCL ranging from 0.156 mg/kg at DU-19 to 0.29 mg/kg at DU-18. Concentrations in subsurface soil (6-36 in. bgs) ranging from 0.070 mg/kg at DU-19 to 0.8 mg/kg at DU-17, and 95 UCL ranging from 0.0804 mg/kg at DU-19 to 0.96 mg/kg at DU-17. Background concentrations for thallium ranged from 0.043 to 0.18 mg/kg, with a 95UCL of 0.215 mg/kg. The EcoSSL for thallium in soil is 0.05 mg/kg (EPA 2018). For reference, the EPA residential RSL for thallium in soil is 0.078 mg/kg (EPA 2021).

- Zinc: concentrations in surface soil (0–6 in. bgs) ranging from 83 mg/kg at DU-18 to 8,200 mg/kg at DU-17, and 95UCL ranging from 145 mg/kg at DU-19 to 10,200 mg/kg at DU-

17. Concentrations in subsurface soil (6-36 in. bgs) ranging from 60 mg/kg at DU-19 to 5,600 mg/kg at DU-18, and 95 UCL ranging from 63.5 mg/kg at DU-19 to 7,850 mg/kg at DU-18. Background concentrations for zinc ranged from 21 to 72 mg/kg, with a 95UCL of 69.1 mg/kg. The EcoSSL for zinc in soil is 46 mg/kg (EPA 2018). For reference, the EPA residential RSL for zinc in soil is 2,300 mg/kg (EPA 2021).

- HMW PAHs: concentrations in surface soil (0–6 in. bgs) ranging from 4.953 mg/kg at DU-17 to 21.51 mg/kg at DU-19, and 95UCL ranging from 11.3 mg/kg at DU-17 to 30.6 mg/kg at DU-19. Concentrations in subsurface soil (6-36 in. bgs) ranging from 0.1511 mg/kg at DU-19 to 8.57 mg/kg at DU-18, and 95 UCL ranging from 0.482 mg/kg at DU-19 to 12.4 mg/kg at DU-18. Background concentrations for HMW PAHs ranged from 0.551 to 9.9 mg/kg, with a 95UCL of 9.59 mg/kg. The EcoSSL for HMW PAHs in soil is 1.1 mg/kg (EPA 2018). For reference, there is no EPA residential RSL for total HMW PAHs.

Note that types of contamination that may be eligible for DERP FUDS response include CERCLA hazardous substances or pollutants or contaminants, military munitions, POL, or unsafe conditions posed by buildings or structures. Metals that are CERCLA hazardous substances include arsenic cadmium, chromium, copper, lead, nickel, selenium, and silver. The full metals list is reported for completeness purposes, however metals that are not a CERCLA hazardous substance (for example, manganese) are not eligible for DERP FUDS response.

10.1.4 Phase I Data Gaps

A summary of data gaps identified following completion of Phase I is presented in the subsections below. Phase II is being conducted to close data gaps identified after completion of Phase I and support decision making. Sampling design and rationale for Phase II is discussed in Worksheets #11 and #17.

10.1.4.1 Bioavailability of Metals and PAHs in Upland and Hydric Soil

Metals and PAH concentrations in upland soil may pose risk to terrestrial ecological receptors (plants and soil invertebrates). The highest concentrations of CERCLA hazardous metals in Phase I upland surface soil were detected at DU-4 (copper at 600 mg/kg and zinc at 72 mg/kg), DU-7 (lead at 2,000 mg/kg), DU-11 (arsenic at 43 mg/kg). In addition, the highest total HMW PAH concentrations (191.85 mg/kg) and total LMW PAH concentrations (63.95 mg/kg) in Phase I soil were reported at DU-13.

Metals and PAH are present in hydric surface soil (0–6 in. bgs) and hydric subsurface soil (6–36 in. bgs) at AOC #16 – Area D (former Dumping Area) at concentrations that may pose risk to ecological receptors (birds, mammals, terrestrial plants, and soil invertebrates). The highest concentrations of CERCLA hazardous metals in Phase I hydric surface soil were detected at DU-17 and DU-18 (specifically lead at 1,200 to 2,900 mg/kg, cadmium at 4.2 to 16 mg/kg, and copper at 83 to 1,700 mg/kg). In addition, the highest total HMW PAH concentrations in Phase I hydric

soil were reported in surface soil at DU-19 (21.51 mg/kg).pose risk to insectivorous birds and mammals.

High levels of metals and PAHs in soil are not necessarily indicative of the adverse effects to human health and/or ecological receptors. The fate and behavior of metals in in soil are determined by retention/release processes, which regulate the concentration of metals in the soil solution and thus their bioavailability. Soil properties, such as pH, texture, ion exchange capacity, soil organic matter content, and porosity influence bioavailability. Natural processes such as adsorption, ion exchange, complexation, and precipitation can cause metals to become immobilized; and therefore, not bioavailable.

The bioavailability of PAHs in soil is influenced by organic carbon quality and quantity, aging and weathering, microbial action, methylation/hydroxylation, adsorption/ desorption hysteresis, and ultra-violent light interaction (Fairbrother 2005). The soil organic carbon-water partition coefficients for PAHs vary depending on grain size, with the highest values in silt (fine particles) followed by sand and clay. PAHs undergo a weathering process such that the lighter chain fractions are removed (primarily by volatilization). Heavier fractions bind more readily to organic matter and remain behind in the soil horizon. In addition, aging reduces the bioavailability of PAHs (Johnson et al. 2002). Chemicals in soils "age" by becoming incorporated inside the crystal lattice structure of the soil particle or partitioning onto organic matter or soil nanopores (Nam et al. 1998), so they are no longer available for uptake by organisms.

Further evaluation is needed to evaluate potential bioavailability of metals and PAHs in upland and hydric soil to terrestrial ecological receptors. Soil characteristics used to determine the distribution of metals in soil and sorption and release processes include pH, grain size, TOC, CEC, and redox potential. Soil characteristics used to evaluate the bioavailability of PAHs include TOC and grain size. pH data were evaluated in onsite soil during Phase I, but additional data (grain size, TOC, CEC, and redox potential) are needed to fully evaluate bioavailability.

10.1.4.2 Contaminant Migration Pathways and Shoreline Exposure Routes

Contaminant migration pathways to the Hudson River and Iona Marsh shorelines were not identified within the upland area (DU-1 through DU-7, DU-11 through DU-14, and DU-22), and therefore evaluation of impacts to the shoreline environment are not recommended for the upland area DUs.

The marsh area between Round and Iona Islands (AOC #16 – Area D [former Dumping Area]) is inundated (under water) during high tide and storm events. During the MMRP intrusive investigation of AOC #16 – Area D (former Dumping Area), free-standing water was encountered during low tide in test pits excavated at depths of 3 to 4 feet (ft) bgs. Defined surface water migration pathways were identified during Phase I leading from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines. These pathways include overland flow with tidal water and stormwater runoff to the Hudson River, a tidal wetland swale that appears

to convey flow to the west toward Iona Marsh, and a riprap filled drainage swale that directs flow south towards Iona Marsh (Figure 10-1).

The surface water migration pathways from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines and impacts to shoreline media (surface water and sediment) were not evaluated during Phase I. AOC #16 – Area D (former Dumping Area) is inundated during high tide and storm events. There is potential for metals within soil at AOC #16 – Area D (former Dumping Area) to be mobilized during tidal inundation and transported via surface water to the Hudson River shoreline. In addition, although water was not observed in the tidal wetland swale and riprap filled drainage swale leading to Iona Marsh during the shoreline reconnaissance, these drainages may convey water and associated contaminants during storm events. Surface water data are needed within AOC #16 – Area D (former Dumping Area), along the Hudson River shoreline, and along defined drainages leading from AOC #16 – Area D (former Dumping Area) to Iona Marsh to evaluate the surface water migration pathway. Additionally, surface water and sediment data are needed to determine if metals related to former DoD activities are present in shoreline media and to evaluate risk to human health and ecological receptors.

10.1.4.3 Background Hudson River Water and Sediment Concentrations

Several metals are naturally occurring in sediment and surface water. In addition, the Hudson River PCB Superfund Site encompasses nearly 200 miles of the Hudson River from Hudson Falls, New York, to the Battery in New York City due to PCB contamination (EPA 2017). This Superfund Site encompasses the shoreline of Iona Island. In addition, the Hudson River is impacted with contaminants from upstream sources, including industrial facilities, as well as other pollutants from urban and agricultural runoff, and sewage and stormwater discharge. Although industrial discharges have been reduced, pollution from urban and agricultural runoff and sewage and stormwater discharge continues, and a legacy of past pollution remains in river sediment. Therefore, shoreline surface water and sediment at Iona Island are likely impacted by contamination from the Hudson River.

Active River Subdivision (CSX Transportation) railroad tracks parallel the western shoreline of the Hudson River. The Iona Marsh Shoreline located along the west side of Iona Island is divided by the railroad embankment. Contaminants associated with the railroad may include metals, PAHs, and pesticides associated with normal railroad operations; petroleum products dropped or spilled from trains including oil, lubricants, and diesel; coal ash from engines; creosote from railroad ties; and PAHs from exhaust. Areas along the railroad tracks are likely to have been affected by normal operation of the rail line, with residual levels of contamination in media adjoining the railroad right-of-way.

Due to the potential for metals to naturally occur in sediment, and due to the potential for shoreline media to be impacted by contaminants in/associated with the Hudson River and/or normal operations of the railroad, regional background/upstream concentrations of metals in surface water and sediment and statistical comparisons of onsite and background metals concentrations are needed to determine whether metals in shoreline media at Iona Island can be attributed to former

DoD activities. Hudson River and railroad impacts to Iona Island shoreline surface water and sediment will be assessed by collecting background samples at Con Hook, located approximately 3 miles upstream of Iona Island on the western side of the Hudson River (Section 11.1.1.4, Section 17.1.3, Section 17.1.4). Con Hook has similar depositional environments to Iona Island, with approximately 7 acres of upland habitat and another 12 acres of a *Phragmites*-dominated tidal marsh (Con Hook Marsh) (Figure I-4). The Active River Subdivision (CSX Transportation) railroad tracks run along the western boundary of the marsh. Similar to Iona marsh, Con Hook Marsh adjoining the railroad right-of-way may be impacted by contaminants associated with normal operation of the rail line.

10.2 PHYSICAL PROFILE

10.2.1 Topography

The upland area of Iona Island has rocky terrain, with varying degrees of slopes. Elevations range from about 0 ft National Geodetic Vertical Datum 1988 (NAVD88) along the along the immediate shoreline to 88 ft NAVD88 in the northern portion of the island.

Area D (former Dumping Area) is confined to a low-lying marsh area between Round and Iona Island. This area is bounded to the northeast by the Hudson River and to the southwest by a fill area emplaced by the Palisades Interstate Park Commission (PIPC) following Navy transfer of the FUDS in 1965 (Figure 10-1). Surface elevations within AOC #16 – Area D (former Dumping Area) range from 0 ft above mean sea level (amsl) at the Hudson River to 2 ft amsl. Elevations within the marsh area rise slightly within the fill area to the southwest, ranging from 2 to 5 ft amsl. Elevations rise steeply along the bedrock wall of Round Island to the southeast and along Iona Island to the northwest.

10.2.2 Surface Water

There is no standing surface water present in the upland area of the island. In addition, no surface water migration pathways (i.e., defined channels, culverts, swales, leading to the Hudson River and Iona Marsh shorelines were identified within the upland area during the Phase I Shoreline Reconnaissance (Figure I-2). Two potential surface water migration pathways were noted during the Phase I shoreline reconnaissance leading from the southwestern portion of the marsh between Round and Iona Islands to Iona Marsh (i.e., AOC #16 – Area D [former Dumping Area]) (Figure 10-1). These pathways include a riprap filled drainage swale that directs flow to the south towards the marsh and a tidal wetland swale that appears to convey flow to the west toward the marsh. Although water was not observed in these drainages during the shoreline reconnaissance, these drainages may convey water during high tide or storm events.

10.2.3 Sediment

Discrete sediment grab samples were collected during the Phase I shoreline reconnaissance to evaluate the physical characteristics of Hudson River and Iona Marsh shoreline sediment (TOC

and grain size) and to evaluate the potential for sediment to accumulate contaminants. Hudson River sediment along the eastern shoreline of AOC #16 – Area D (former Dumping Area) and Iona marsh shoreline sediment at the outlets of the riprap filled drainage swale tidal wetland swale were evaluated during Phase I (Figure 10-1).

Hudson River sediment along the eastern shoreline of AOC #16 – Area D (former Dumping Area) consists of fine-grained sand (21.89 to 80.69 percent) and medium-grained sand (15.34 to 36.45 percent) with lesser amounts of coarse-grained sand (1.08 to 14.84 percent) and gravel (2.89 to 22.73 percent), and trace amounts of silt and clay (1.16 to 2.94 percent). Hudson River shoreline sediment was classified as SP (poorly graded sand) using the using the Unified Soil Classification System [USCS] (ASTM 2006). TOC content ranged from 4,700 to 31,000 mg/kg.

Iona marsh shoreline sediment at the outlet of the riprap filled drainage swale consists of silt (82.64 percent), with lesser amounts of fine sand (9.13 percent), clay (5.01 percent), and medium-grained sand (3.22 percent). Sediment was classified as ML (silt) using the USCS (ASTM 2006). TOC content was reported at 110,000 mg/kg.

Iona marsh shoreline sediment at the outlet of the tidal wetland swale consists primarily of silt (80.25 percent), with lesser amounts of clay (9.23 percent), fine sand (6.57 percent), and medium-grained sand (3.94 percent). Sediment was classified as ML (silt) using the USCS (ASTM 2006). TOC content was reported at 62,000 mg/kg.

10.2.4 Soil

Soil encountered across the majority of upland area during Phase I was shallow, with a depth to hand auger refusal ranging from 6 to 12 in. bgs. Deeper soil in the upland portion of Iona Island was encountered at DU-20 and DU-21, where hand auger refusal was encountered at 18 and 24 in. bgs, respectively) (Figure I-2). Surface soil types in upland DUs were classified primarily as brown, dry silt with varying amounts of organic material and trace amounts of coarse-grained material (ranging from very fine sand to coarse gravel). Surface soil was classified as ML (silt) and OL (organic silt) using the USCS (ASTM 2006). Subsurface soil types in upland DUs (i.e., DU-20 and DU-21) was classified as brown, dry silt, with trace fine- to coarse-grained sand and fine- to coarse-grained gravel (ML) (ASTM 2006).

Hydric soil is present at AOC #16 – Area D (former Dumping Area) (DU-17, DU-18, and DU-19) (Figure 10-2). This area is inundated during high tide and storm events. During low tide, free-standing water is encountered at approximately 3-4 ft bgs. Surface soil at DU-17, DU-18, and DU-19 was classified as dark brown, moist, moderately to well sorted silt with organic material and trace fine-grained sand (ML and OL). Subsurface soil varied from black, very moist, poorly sorted sand (SW) at DU-17 to dark brown to dark grey (with a bluish tint), well sorted silt with trace fine-grained sand (ML) (DU-18 and DU-19). Burn pit material and debris was noted in test pits excavated during the MMRP intrusive investigation. This material potentially originates from a former incinerator (AOC #2 – Former Building 121) that was located near the Hudson River shoreline immediately north/northwest of AOC #16 – Area D (former Dumping Area) within

DU-17. The base of burn pit material was not determined during the MMRP RI as test pits were terminated at approximately 4 ft bgs due to water inundation.

10.2.5 Hydrogeology

Groundwater was not encountered in upland areas during Phase I soil sampling conducted in March and April 2020. Moist to very moist surface and shallow subsurface soil was encountered at AOC #16 – Area D (former Dumping Area) (DU-17, DU-18, and DU-19). In addition, saturated soil was encountered at AOC #16 – Area D (former Dumping Area) at approximately 3 to 4 ft bgs during intrusive investigations conducted under the MMRP RI in August 2020. AOC #16 – Area D (former Dumping Area) is influenced by the Hudson River and inundated during high-tide and storm events.

10.3 HTRW SOURCE AREAS

The upland area and AOC #16 – Area D (former Dumping Area) have been retained as general areas of investigation for Phase II due to widespread metals and PAH concentrations detected in Phase I surface soil (0–6 in. bgs) above both EPA screening criteria and background concentrations. Source areas for PAHs and metals are the AOCs investigated during Phase I. Bioavailability of metals and PAHs was not evaluated during Phase I.

AOC #16 – Area D (former Dumping Area) has been retained as potential source area for further investigation in Phase II. Metals were detected in Phase I surface soil (0–6 in. bgs) and subsurface soil (6–36 in. bgs) at DU-17, DU-18, and DU-19, established to evaluate AOC #16 – Area D (former Dumping Area), at elevated concentrations. Surface water and potential contaminant migration pathways leading from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines were noted during Phase I.

10.4 CONTAMINANTS OF POTENTIAL CONCERN

The list of contaminants of potential concern (COPCs) identified for Phase II media consists of the following:

- CERCLA hazardous metals in surface water and sediment along the shorelines of AOC #16 – Area D (former Dumping Area) (specifically, arsenic, cadmium, chromium, cobalt, copper, lead, mercury, nickel, and zinc)

10.5 CONCEPTUAL SITE MODEL

Media of concern and human and ecological receptors were evaluated to identify potential exposure routes linking the two and determine which pathways are complete and significant. The CSM is used to identify all complete, potentially complete, or incomplete exposure pathways to potential current and future human and ecological receptors based upon the site history and current

and anticipated future site use. Figures 10-2 and 10-3 present the preliminary Phase II CSMs for human health and ecological receptors.

10.5.1 Release Mechanisms

The primary mechanism includes spills, direct releases, and dumping, as presented in the UFP-QAPP (EA 2020a). Secondary release mechanisms include soil removal/reworking, infiltration/leaching, and runoff/erosion. Buildings were demolished and soil was reworked following Navy transfer of the FUDS property to PIPC in 1965. Contaminants in surface and subsurface soil were likely mobilized beyond the footprint of the AOCs during demolition of former buildings/structures and reworking of surface and subsurface soil. Potential contaminants may have leached/infiltrated into the subsurface through gravity and/or percolation of precipitation/surface water, thus migrating to the subsurface soil and groundwater; leaching/infiltration may be ongoing if a soil source is present at Iona Island, particularly at AOC #16 - Area D (Dumping Area). In addition, contaminants may have been transported along the surface via overland runoff and erosion and ultimately deposited in shoreline sediments; these release mechanisms may be historically significant but are unlikely to contribute to current exposure pathways due to the vegetated nature of the soil.

Constituents released through deposition may have remained in soil by adsorbing to soil particles. When released to the environment, constituents such as some metals and PAHs tend to sorb to soil. Contaminants are released from the soil surfaces in the liquid phase in response to changes in the chemical environment of the soil solution. Desorption would be dependent on the solubility of the individual chemical and the properties of the soil.

10.5.2 Contaminant Fate and Transport

10.5.2.1 Upland Area Soil (DU-1 through DU-7, DU-9 through DU-16, and DU-22)

Surface water migration pathways were not identified leading from upland area surface soil to the shoreline. The upland area is extensively vegetated, which limits potential for contaminant migration via soil erosion and stormwater runoff. In addition, groundwater was not encountered in upland areas during Phase I.

10.5.2.2 AOC #16—Area D (former Dumping Area) (DU-17, DU-18, and DU-19)

Potential contaminant pathways leading from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines were identified during Phase I. AOC #16 – Area D (former Dumping Area) is tidally influenced and inundated during high tide and storm events. Water was encountered at AOC #16 – Area D (former Dumping Area) during low tide at a depth of 3–4 ft bgs. This water rises during high tide and storm events and inundates AOC #16 – Area D (former Dumping Area).

Metals constituents with high solubility at AOC #16 - Area D (former Dumping Area) may be mobilized during tidal and storm water inundation, transported to the Hudson River via advective transport (bulk movement) or as particulate complexes, and ultimately deposited in Hudson River shoreline sediment. In addition, potential pathways leading from AOC #16 – Area D (former Dumping Area) to Iona Marsh were observed during the shoreline reconnaissance, including a riprap filled drainage swale and tidal drainage swale. Although water was not observed in these drainages during the shoreline reconnaissance, these drainages may be inundated during storm events and may provide an intermittent discharge pathway for flood water within AOC #16 – Area D (former Dumping Area) to Iona Marsh.

The types of constituents detected in site soil at AOC #16 – Area D (former Dumping Area) would be bound to soil particles and unlikely to desorb (leach) from soil to water. In addition, the phragmites root system would limit advective transport (bulk movement) of soil and contaminants. Although release and transport mechanisms are unlikely to contribute to current exposure pathways due to the vegetated nature of the soil at AOC #16 – Area D (former Dumping Area), release and transport mechanisms may be historically significant.

Shoreline sediment consisting of fine-grained particles and high organic matter (i.e., TOC) are more likely to present a “sink” for historical constituents and serve as a source of exposure to organisms. Sediment consisting of courser-grained particles (e.g., sand and gravel) with low TOC are less likely to accumulate historical constituents. Based on the fine-grained nature and high TOC content of Iona Marsh shoreline sediment samples, sediment in the marsh appears conducive for accumulation of constituents. Marsh sediment may therefore have accumulated historical constituents from AOC #16 – Area D (former Dumping Area) that were transported to the shoreline via advective transport (bulk movement) along surface water migration pathways (riprap filled drainage swale and tidal wetland swale).

Based on the coarser grain size and low TOC content of Hudson River shoreline sediment samples, these sediments are less likely to accumulate historical constituents. In addition, contaminant concentrations transported to the Hudson River shoreline would be reduced through dilution and dispersion in Hudson River water.

10.5.3 Nature and Extent of Contamination

10.5.3.1 Upland Area Soil (DU-1 through DU-7, DU-11 through DU-16, and DU-22)

Elevated concentrations of metals and PAHs are widespread throughout the upland area of Iona Island (DU-1 through DU-7, DU-11 through DU-16, and DU-22) (Figure I-2). The highest concentrations of CERCLA hazardous metals in Phase I surface soil were detected at DU-4 (copper at 600 mg/kg and zinc at 72 mg/kg), DU-7 (barium at 110 mg/kg and lead at 2,000 mg/kg), and DU-11 (arsenic at 43 mg/kg. In addition, the highest PAH concentrations in Phase I soil were reported at DU-13 (total HMW PAH concentration of 191.85 mg/kg and total LMW PAH concentration of 63.95 mg/kg).

10.5.3.2 AOC #16 - Area D (former Dumping Area) (DU-17, DU-18, and DU-19)

Fill material and impacted soil at AOC #16 – Area D (former Dumping Area) is confined to the low-lying marsh area between Round and Iona Island (Figure 10-1). The northeastern boundary of AOC #16 – Area D (former Dumping Area) is the Hudson River shoreline. Two surface water drainages (riprap filled drainage swale and tidal wetland swale) are present at the southwestern boundary of AOC #16 – Area D (former Dumping Area). The highest metals concentrations were reported in soil at DU-17 and DU-18, with concentrations decreasing in soil at DU-19.

The surface elevation of AOC #16 – Area D (former Dumping Area) (0–2 ft amsl) is the same as the Hudson River shoreline along the northeastern boundary of AOC #16 – Area D (former Dumping Area). This was confirmed during field observations when it was noted that the majority of the inland portion of AOC #16 – Area D (former Dumping Area) is inundated during high tide. Based on a review of the depth chart of the Hudson River near AOC #16 – Area D (former Dumping Area) and visual observations of the shoreline adjacent to AOC #16 – Area D (former Dumping Area) during the Phase I Shoreline Reconnaissance and the MMRP digital geophysical mapping shoreline survey, there is a shallow bedrock shelf extending out from AOC #16 – Area D (former Dumping Area) into the Hudson River. Based on the Hudson River bathymetry (USACE New York District 2018), AOC #16 – Area D (former Dumping Area) topography (EA 2020b), and field observations, the bedrock elevation at the Dump Area is likely to be the same as the elevation of the near shore bedrock shelf. This elevation results in an estimated bedrock depth of 5 ft bgs, which would also be the maximum depth of AOC #16 − Area D (former Dumping Area) fill.

10.5.4 Exposure Media

The primary terrestrial media of concern for human health and ecological receptors in the upland area is soil. Groundwater was not encountered in the upland area during Phase I. Contaminant transport pathways from upland soil to the shoreline were not identified during Phase I, and therefore shoreline media are unlikely to be impacted by contaminants from the upland area.

The primary terrestrial media of concern for human health and ecological receptors in the marsh area between Iona and Round Islands (AOC #16 – Area D [former Dumping Area]) is soil. However, AOC #16 – Area D (former Dumping Area) is inundated during high tide and storm events, and soil is covered by a dense mat of phragmites, limiting potential use and exposure by human receptors and terrestrial ecological receptors (soil invertebrates and insectivorous birds and mammals).

Surface water migration pathways to the shoreline have been identified at AOC #16 – Area D (former Dumping Area). There is potential for constituents to be present in shoreline media (surface water and sediment) at concentrations that pose risk to human health and aquatic ecological receptors.

Sediments are ingested by benthic invertebrates, benthic forage fish, and incidentally by mammals and birds that feed in the aquatic environment. Aquatic and wetland plants have root systems that are also directly exposed to sediment. Areas with heavy *Phragmites* root mats will limit wildlife and invertebrate exposure to sediment and these areas will not be sampled. Desorption of contaminants from sediment to the water column can occur, impacting water column invertebrates (i.e., zooplankton) and fish.

Aquatic-feeding wildlife (e.g., birds and mammals) may ingest constituents through the prey items that they consume as food. For instance, insectivorous birds consume benthic invertebrates; herbivorous birds and mammals consume wetland aquatic plants; large piscivorous mammals (e.g., river otter) and birds (e.g., bald eagle and heron) consume fish. Ingestion of constituents in sediment, surface water, and aquatic biota are considered potentially complete exposure pathways for aquatic wildlife.

10.5.4.1 Human Health Exposure

Iona Island is part of the Hudson River National Estuarine Research Reserve and is under the administration of PIPC. The majority of the Island is open space. Park staff use the Island for storage. Bear Mountain State Park employees access buildings used as storage areas at the property. The site recreational use is limited to educational outings and history tours (6-8 times per year with approximately 50 people). Additionally, the island has been historically used for camping. The Boy Scouts have used the Site within the past 5 years for activities. In addition, researchers are presently working in the marshes. Potential trespassers are a possibility because the shoreline is not fenced. In addition, because the shoreline is not fenced, residents who live near the Island may access the site routinely during boating season.

The future use of Iona Island is anticipated to remain consistent with current use and will continue to be maintained by park employees and be used as an area for research. Hypothetical future plans for Iona Island may include recreational use and construction of a nature or retreat center; however, these plans are not currently approved. Communication with the Director of Development and Special Projects at PIPC indicates that construction or redevelopment is unlikely at the site for the foreseeable future.

The Phase I investigation evaluated exposure to onsite soil. Based upon the current use and reasonably anticipated future use of the Iona Island, the following receptors were identified for onsite soils:

- Bear Mountain State Park employees (park personnel)
- Researchers
- Construction Worker
- Recreational User
- Trespassers
- Boy Scout/Camper.

Concentrations of lead within AOC #16 – Area D (former Dumping Area) (specifically, DU-17 and DU-18) may pose risk to human health receptors. Complete and/or potentially complete exposure pathways identified for these receptors include the following (Figure 10-3):

- Incidental ingestion of soil
- Dermal contact with soil
- Inhalation of windblown particulates from soil.

Exposure to shoreline surface water and sediment is being evaluated during Phase II. The receptors selected for evaluation of Phase II media of concern include:

- Bear Mountain State Park employees (park personnel)
- Researchers
- Recreational User
- Trespassers
- Boy Scout/Camper.

Currently, park personnel are present on the island, and these personnel may contact the Hudson River and Iona Marsh shoreline sediment and surface water within the migration pathways identified from AOC #16 – Area D (former Dumping Area). Additionally, researchers are also present on the island and would likely contact sediment and surface water within the migration pathways identified from AOC #16 – Area D (former Dumping Area).

Currently, there are no barriers to restrict access to the shoreline of the Island. There is a potential for kayakers or other boaters to visit the site along the shoreline from the Hudson River. These receptors are identified as recreational users that represents a multiple age-range family that may visit the site. The adult recreational user is assumed at over 18 years of age. The adolescent recreational user is assumed from 6 to 16 years. The child recreational user is assumed from 2 to 6 years. Recreational users younger than 2 years of age are not anticipated due to site access and site conditions that are not conducive to playgrounds or other play areas. The recreational user is representative of other low frequency receptors that may visit the site during tours or other events. Therefore, the assessment of the recreational user is protective of other low frequency visitors. The recreational user may contact sediment and surface water within the migration pathways identified from AOC #16 – Area D (former Dumping Area).

Trespassers are also potential receptors due to no access controls along the shoreline. The trespasser is assumed to be an adolescent 12 to 18 years of age and represents a slightly older adolescent who may visit the site while boating along the eastern shoreline (Hudson River) or western shoreline (Iona Marsh) or hiking in the state park.

The Boy Scout/Camper are also potential receptors to surface water and sediment within the migration pathways identified from AOC #16 – Area D (former Dumping Area). This receptor is

assumed at an age from 6 to 16 years who would visit the site periodically to camp and may perform other activities, such as planting or cleanup.

The following exposure pathways are considered potentially complete for Phase II (Figure 10-3):

- Incidental ingestion of sediment and/or surface water
- Dermal contact with sediment and/or surface water.

None of the receptors are expected to inhale windblown particulate from sediment because the exposure route assumes particulates become airborne through wind action. Due to the moisture content of sediment, particulates are not expected to become airborne.

10.5.4.2 Aquatic Ecological Exposure

The Phase I investigation evaluated onsite soil. Terrestrial ecological receptors selected for evaluation are discussed in the UFP-QAPP (EA 2020a). Based on the ecological setting and media of concern, ecological receptors for the Phase II investigation include aquatic organisms such as plants, aquatic life, benthic invertebrates, and aquatic-feeding mammals and birds. Aquatic ecological receptors selected for evaluation to media of concern (sediments and surface water) at Iona Island include the following: aquatic plant community, benthic invertebrate community, water column invertebrates (zooplankton community), forage fish, predatory fish, herptiles, birds (mallard duck, lesser scaup, great blue heron) and mammals (muskrat and river otter). Additional details regarding the rationale for selection and information on how these organisms will be evaluated is presented in Section 10.5.4.2.4.

Media of concern (sediment and surface water) and ecological receptors are evaluated to determine potential exposure routes linking the two, and to evaluate which pathways are complete and significant. The sections below identify the major routes of exposure and their applicability to each receptor group. A diagram depicting complete and incomplete exposure pathways, exposure media, and receptors is provided as Figure 10-4.

10.5.4.2.1 Sediment Exposure

Plants, benthic invertebrates, and small benthic forage fish may be exposed to constituents in sediment through direct contact and ingestion. Plants may absorb chemicals from sediment via their roots. Emergent plants may also adsorb chemicals from air or airborne particles through their leaves, although the waxy surfaces of leaves limit this exposure.

Benthic invertebrates and small forage fish may be exposed to constituents in sediment through direct contact and ingestion. Small fish include minnows and juvenile stages of larger predatory fish. The top 6 inches of sediment generally contain the highest amount of organic matter, which provides a source of food for invertebrates, and fish. In addition, porewater, the interstitial water located between sediment particles, is directly ingested by infaunal invertebrates through their gills. Plant roots can also uptake porewater. Porewater is a potentially complete exposure pathway

because constituents may desorb from sediments; however, it will not be evaluated quantitatively. Exposure pathways linking plants, benthic invertebrates, and forage fish to sediment are complete and relevant for assessment.

Wildlife (birds and mammals) and herptiles may also ingest constituents in sediment by incidental ingestion while grooming or foraging. Most wildlife have protective outer coverings such as fur or feathers that prevent or limit the dermal absorption of chemicals from environmental media (U.S. Army Center for Health Promotion and Preventative Medicine [USACHPPM] 2004). EPA guidance identifies that, in most cases, dermal exposures are likely to be less significant than exposures through ingestion, and their evaluation involves considerable uncertainty (USACHPPM 2004). This exposure route is considered potentially complete because it is relatively insignificant for wildlife.

10.5.4.2.2 Surface Water Exposure

Water column invertebrates, also known as zooplankton, inhabit the water column and provide a base of the food web for larger aquatic life. Zooplankton can include larval forms of larger aquatic life or organisms like water fleas (*Daphnia spp*.) which spend their entire lives in the water column. These organisms are in direct contact with surface water and ingest the water through their gills.

Large predatory fish (e.g., bass, carp) that inhabit the water column also ingest and are in direct contact with surface water. These large fish are less exposed to sediments than smaller forage fish because they require deep water to thrive. Exposure pathways linking water column invertebrates, forage fish, and predatory fish are complete and relevant for assessment. Wildlife (birds, mammals) and herptiles are also exposed to surface water, primarily through ingestion. As noted above, wildlife have protective outer coverings such as fur or feathers that prevent or limit the dermal absorption of chemicals, therefore, dermal exposures are likely to be less significant than exposures through ingestion and this pathway is considered potentially complete.

10.5.4.2.3 Food Web Exposure

The most significant exposure route for aquatic birds and mammals is ingestion of constituents in contaminated food items, which includes lower trophic level aquatic biota (plants, invertebrates). Since some constituents can bioaccumulate in tissue, birds and mammals ingest constituents through the food web. Ingestion of constituents in sediment, surface water, and aquatic biota are considered complete and significant exposure pathways for birds and mammals.

10.5.4.2.4 Aquatic Ecological Receptors

The proposed aquatic receptors for evaluation are based on feeding guilds and availability of wildlife exposure models. The aquatic receptors selected for evaluation include the following:

- The likelihood of a species to use the site and the area immediately surrounding it

- The potential for exposure to constituents based on the feeding habits and life history of the organisms/feeding guild represented by the receptor species
- Life history and exposure information for the selected receptor species
- Toxicity information for the representative receptor species
- The societal importance of the species (e.g., threatened and/or endangered species, species of special concern).

The rationale for selection of representative receptor species and information on how each receptor group will be evaluated is provided below:

Plant Community

Based on the general nature of available plant toxicity data, no specific aquatic plant species are selected for evaluation. Instead, the assessment evaluates the potential for adverse effects to aquatic plant communities. There are no screening values protective of aquatic plants; therefore, plant communities are evaluated by comparing chemical concentrations in sediment and surface water to sediment and surface water benchmarks.

Benthic Invertebrate Community

Based on the general nature of available benthic invertebrate toxicity data, no specific species are selected for evaluation. Instead, the assessment evaluates the potential for adverse effects to benthic invertebrate communities. Benthic invertebrate communities are evaluated by comparing chemical concentrations in sediment to sediment quality benchmarks. Although benthic invertebrates may be exposed to surface water and porewater, sediment is considered the primary complete exposure pathway to be evaluated quantitatively.

Zooplankton Community

Based on the general nature of available invertebrate toxicity data, no specific species are selected for evaluation. Instead, the assessment evaluates the potential for adverse effects to water column invertebrate communities. Zooplankton communities are evaluated by comparing chemical concentrations in surface water to relevant surface water quality criteria.

Fish Community

Based on the general nature of available fish toxicity data, no specific species are selected for evaluation. Instead, the assessment evaluates the potential for adverse effects to forage and predatory fish communities. Forage fish communities are evaluated by comparing chemical concentrations in surface water and sediment to surface water quality criteria and sediment

benchmarks. Predatory fish communities are evaluated by comparing chemical concentrations in surface water only to relevant surface water quality criteria.

Herbivorous Wildlife

Populations of herbivorous mammals will be represented by muskrat (*Ondatra zibethicus*). These rodents spend most of their lives in or near bogs, marshes, lakes, or streams. They are primarily herbivorous and feed upon roots, shoots, bulbs, tubers, stems, and leaves of aquatic plants. They construct dens in riverbanks or along shores of waterways and have a small home range (EPA 1993). Herbivorous mammals will be evaluated by calculating a daily dose of chemical from surface water, sediment, and aquatic biota (aquatic plants). The daily dose will be compared to toxicity reference values for mammals.

Populations of herbivorous birds will be represented by mallard duck (*Anas platyrhynchos*) which are common dabbling (surface-feeding) ducks of freshwater and saltwater wetlands. They feed by dabbling and tipping up in shallow water, often filtering through soft sediments for food. They primarily feed on seeds, leaves, buds, stems, roots, and tubers of aquatic plants (EPA 1993). Herbivorous birds will be evaluated by calculating a daily dose of chemical from surface water, sediment, and aquatic biota (aquatic plants). The daily dose will be compared to toxicity reference values for birds.

Insectivorous Wildlife

Populations of insectivorous birds will be represented by lesser scaup (*Aythya affinis*) which is a common diving duck that primarily consumes invertebrates from the water column and surfaces of aquatic plants and other substrates. Common prey include snails, clams, water fleas, amphipods, midges, chironomids, and leeches. When seeds are abundant, they may also be consumed in large quantities (EPA 1993). Insectivorous birds will be evaluated by calculating a daily dose of chemical from surface water, sediment, and aquatic biota (benthic invertebrates). The daily dose will be compared to toxicity reference values for birds.

Piscivorous Wildlife

Populations of piscivorous mammals will be represented by river otter (*Lontra canadensis*) which are found in freshwater, estuarine, and some marine environments from coastal areas to mountain lakes. They inhabit food-rich coastal areas such as lower portions of streams and rivers, estuaries, non-polluted waterways, lakes and tributaries that feed rivers and areas of little human impact. The bulk of their diet is fish, but they feed on a variety of prey depending on availability and ease of capture. They may also consume crustaceans, insects, amphibians, and reptiles (EPA 1993). Piscivorous mammals will be evaluated by calculating a daily dose of chemical from surface water, sediment, and aquatic biota (forage fish). The daily dose will be compared to toxicity reference values for mammals.

Populations of piscivorous birds will be represented by great blue heron (*Ardea herodias*). These birds have long legs, necks, and bills that are adapted for wading in shallow water and stabbing prey. Most heron species build their nests in trees near their foraging habitat. Herons range in size from the least bittern to the great blue heron (EPA 1993). Great blue heron are assumed to consume small benthic forage fish. Piscivorous birds will be evaluated by calculating a daily dose of chemical from surface water, sediment, and aquatic biota (forage fish). The daily dose will be compared to toxicity reference values for birds.

The least bittern (*Ixobrychus exilis*) is a protected species under the Migratory Bird Act and habitat for the species has been noted on the island. However, due to the lack of an available food web exposure model for the bittern, the species will be represented by the great blue heron, which is a similar wading, piscivorous heron species that shares similar habitats and feeding behaviors. Both birds have long necks and spear-like bills. They feed primarily on aquatic life (e.g., fish) and are common along the margins of waterbodies and wetlands. Their long legs, necks, and bills are adapted for wading in shallow water and stabbing prey. The least bittern is the smallest of the wading birds, whereas the great blue heron is the largest (EPA 1993).

The bald eagle (*Haliaeetus leucocephalus*)is a state and federal protected species that roosts along the Hudson River shoreline on Iona Island, especially during the winter months. Bald eagles are primarily opportunistic, consuming a variety of prey including ducks and birds or whatever prey is available and easiest to obtain (including carrion). They also consume dead and dying fish when available but will capture live fish swimming near the surface or fish in shallow water (EPA 1993). Due to the large home range and lack of site fidelity, the bald eagle will not be evaluated as part of the ecological risk assessment. However, evaluation of great blue heron, another piscivorous species with a smaller home range and more site fidelity, will be protective of bald eagles (EPA 1993).

Herptiles

Reptiles and amphibians could inhabit both terrestrial and aquatic (marsh) areas and be exposed to both sediments and surface water. However, due to the lack of toxicological information, exposure factors, and ecological screening values for these species, only a qualitative evaluation of potential risks is possible. Risks are assumed to be similar to other species evaluated.

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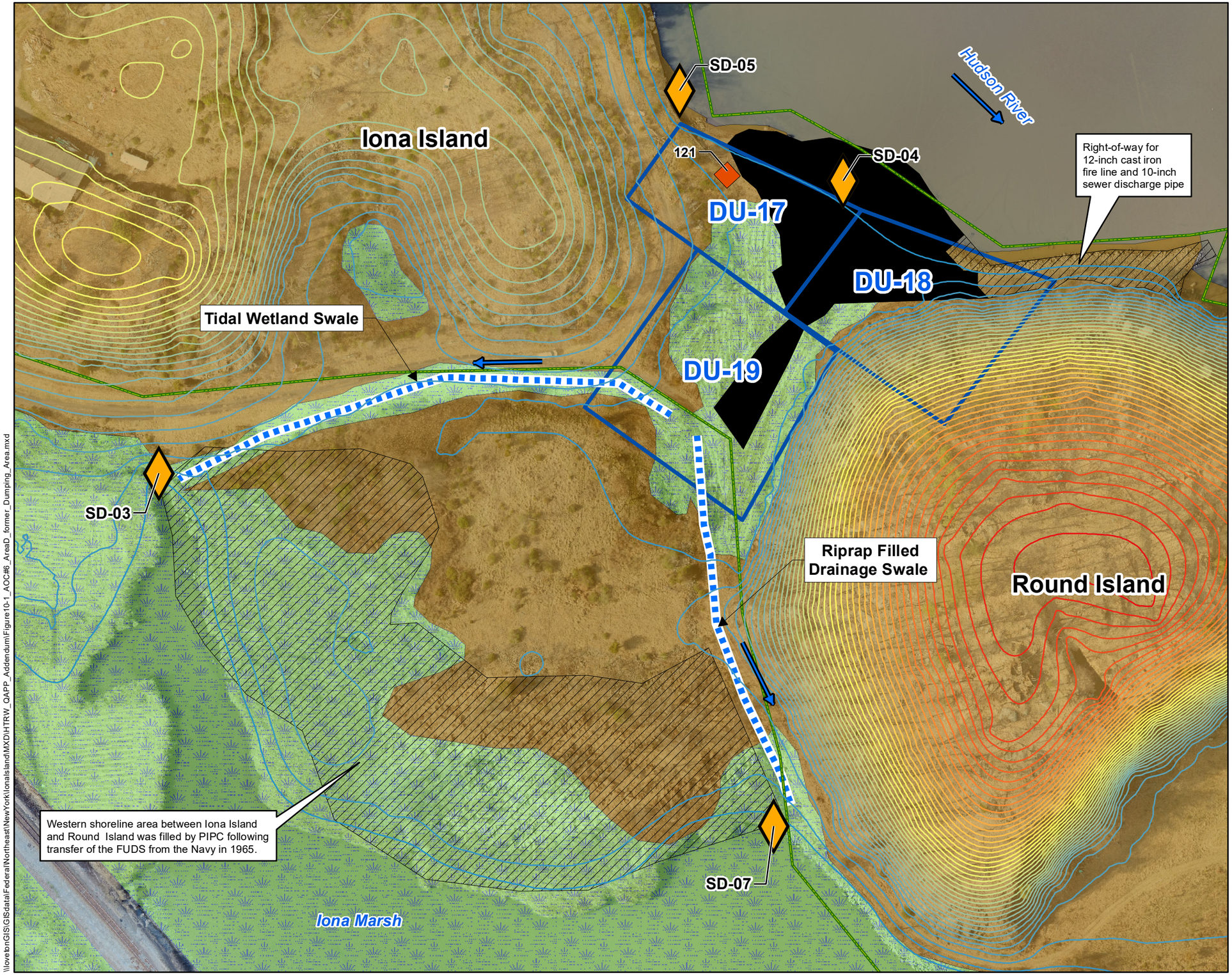
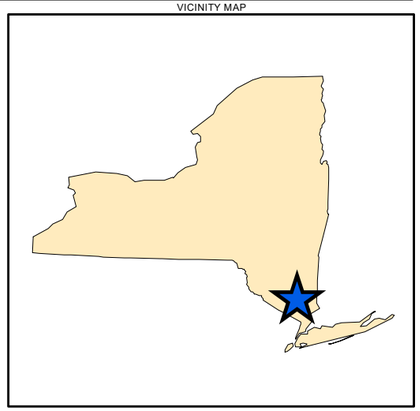


VICINITY MAP



Legend

- FUDS Boundary
- Upland
- Marsh/Wetland
- AOC #16 – Area D (former Dumping Area)
- Bldg 121 Incinerator
- Phase I Soil Decision Unit
- Disturbed Ground/Fill Area
- Surface Water Migration Pathway
- Phase I Sediment Sample Location
- Surface Water Flow Direction
- 2-Foot Topographic Contour Gradations (Feet above mean sea level (amsl)
  - High : 84 ft amsl
  - Low : 0 ft amsl

0 100 200
Feet
1 in = 100 feet

N

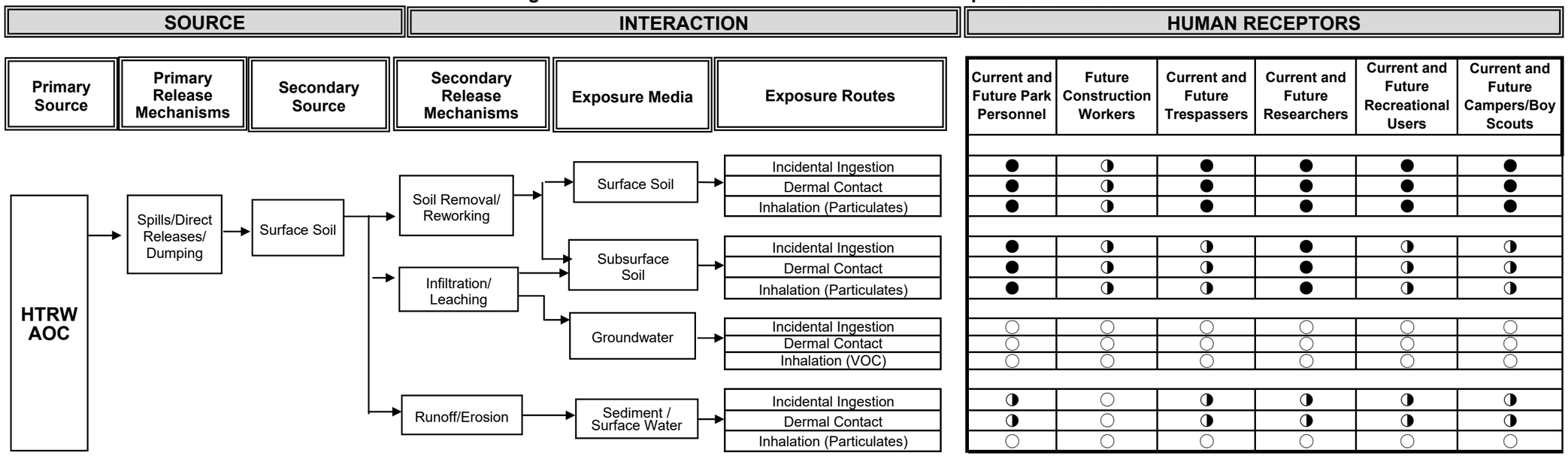
FIGURE 10-1
AOC #6 - Area D (Former Dumping Area) DU-17, DU-18, and DU-19

HAZARDOUS TOXIC AND RADIOACTIVE WASTE QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial and Topographic Contours: EA Engineering, Science, and Technology. April, 7 2020
Disturbed Ground/Fill Area and AOC Boundary: USAGC 2018
Upland and Marsh/Wetland: 2020 EA Shoreline Reconnaissance
Map Date: 1/31/2022

\\lovetongis\GIS\data\Federal\Northeast\NewYork\IonaIsland\MXD\HTRW_QAPP_Addendum\Figure10-1_AOC#6_AreaD_former_Dumping_Area.mxd

Figure 10-2 Human Health Risk Assessment Conceptual Site Model



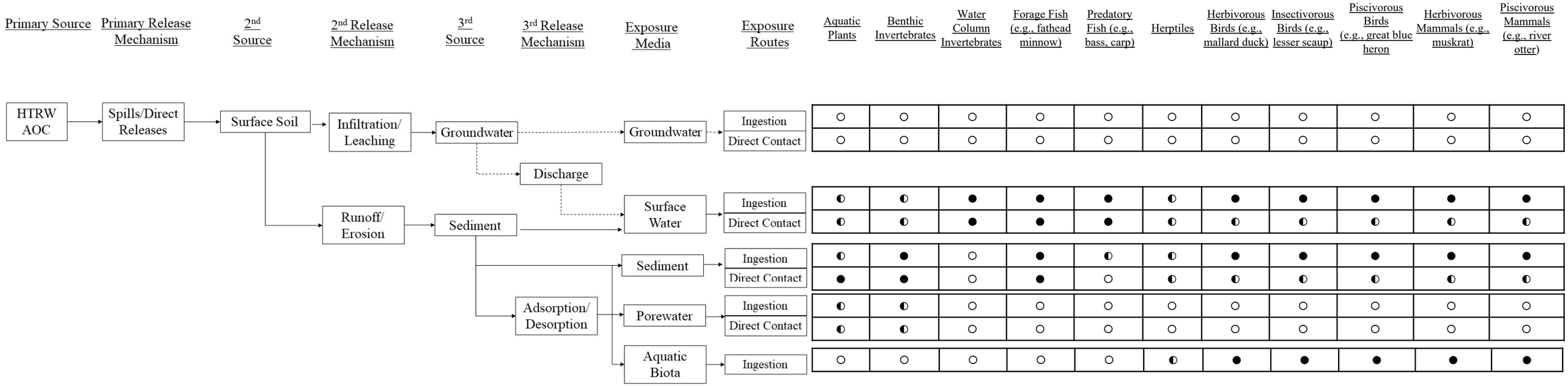
LEGEND

- ● Complete Pathway; confirmed receptor and media interaction (i.e., a source, migration route, and receptor have been identified).
- ◑ Potentially Complete Pathway; receptor and media interaction; however, the exposure may be limited, either due to the type of receptor or the nature of the source, and has not been confirmed.
- ○ Incomplete Pathway; no receptor and media interaction. No evaluation is conducted.

NOTES

Surface soil = defined as the interval from 0 to 6 inches bgs.
Subsurface soil = defined as the interval from 6 to 36 inches bgs.
VOC = Volatile Organic Compound

Figure 10-3 Aquatic Ecological Risk Assessment Conceptual Site Model



LEGEND

- ● Complete Pathway; confirmed receptor and media interaction (i.e., a source, migration route, and receptor have been identified).
- ◐ Potentially Complete Pathway; there is potential receptor and media interaction that may be anticipated, but is not quantified.
- ○ Incomplete Pathway; no receptor and media interaction. No evaluation is conducted.

NOTES

Surface soil = defined as the interval from 0 to 6 inches bgs.
Subsurface soil = defined as the interval from 6 to 36 inches bgs.
VOC = Volatile Organic Compound

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UFP-QAPP ADDENDUM #11 – PROJECT QUALITY OBJECTIVES

This worksheet is used to develop and document project DQOs for Phase II using a systematic planning process that follows the EPA DQO process and documents the environmental decisions that need to be made and the level of data quality needed. The DQO process is outlined in EPA's 2006 guidance document, *Guidance on Systematic Planning Using the DQOs Process* (EPA/240/B-06/001, February 2006) (EPA 2006). The specific QA/QC requirements developed for the site are consistent with those presented in the DoD QSM, Version 5.3 (DoD 2019).

DQOs are both qualitative and quantitative statements that define the type, quality, and quantity of data necessary to support the decision-making process during project activities. The objective of this UFP-QAPP Addendum is to establish standard procedures for Phase II so that the integrity, accuracy, precision, completeness, and representativeness of Phase II collected samples are maintained, and the required DQOs are achieved.

The DQO process provides a systematic procedure for defining the criteria that a data collection design should satisfy. The DQO process, established by EPA and incorporated into the 2012 UFP-QAPP guidance, consists of seven steps that are used during the planning of the data collection process to ensure that field and analytical activities, and the resulting data, meet the project objectives.

11.1 DATA QUALITY OBJECTIVES – 7 STEP PROCESS

The DQOs were developed using the following 7-step process, which is the process designed to: (1) clarify study objectives and decisions to be made based on the data collected (Steps 1 and 2), (2) define the most appropriate type of data to collect (Steps 3 and 7), (3) determine the most appropriate conditions for collecting the data (Steps 4, 5, and 7), and (4) specify acceptable decision error limits based on the consequences of making an incorrect decision (Step 6). The project DQOs are presented below.

11.1.1 State the problem (concisely describe the problem to be studied)

Multi-media sampling is needed to close data gaps identified after completion of Phase I. Phase II is being conducted to further evaluate contaminant migration pathways to the shoreline environment, evaluate potential impacts to ecological receptors, and support development of the HHRA and ERA.

The preliminary COPCs for Phase II are based on the results of Phase I:

- CERCLA hazardous metals in surface water within and sediment along the shoreline of AOC #16 – Area D (former Dumping Area) (specifically, arsenic, cadmium, chromium, cobalt, copper, lead, mercury, nickel, and zinc) (Figure 10-1)

11.1.1.1 Bioavailability of Metals and PAHs in Upland and Hydric Soil

The bioavailability of metals and PAHs in onsite upland and hydric soil was not evaluated during Phase I. Although pH data were evaluated in onsite upland and hydric soil during Phase I, additional physical parameter data (grain size, TOC, CEC, and redox potential) are needed to evaluate bioavailability of metals and PAHs to terrestrial receptors as part of the Refined Screening-Level Ecological Risk Assessment (RSLERA).

11.1.1.2 AOC #16 – Area D (former Dumping Area) Contaminant Migration Pathways and Shoreline Exposure Routes

Contaminant migration pathways from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines were identified during Phase I (Figure 10-1). These pathways include transport to the Hudson River shoreline during high tide and storm events, and transport to Iona Marsh via the riprap filled drainage swale and tidal wetland swale. The contaminant migration pathways and potential impacts to shoreline media (surface water and sediment) were not evaluated during Phase I. Sampling of surface water data are needed to evaluate the potential for offsite migration of metals (including mercury) to the Hudson River and Iona Marsh. In addition, evaluation of surface water and sediment along the Hudson River shoreline of AOC #16 – Area D (former Dumping Area) and at outlets of drainages to Iona Marsh is needed to determine if metals related to former DoD activities are present in shoreline media at concentrations that pose potential significant risk to human health and/or ecological receptors.

11.1.1.3 Background Hudson River Water and Sediment Concentrations

Due to the potential for metals to naturally occur in sediment, and due to the potential for shoreline media to be impacted by contaminants in the Hudson River and/or normal operations of the railroad that parallels the western shoreline of the river, regional background/upstream concentrations of metals in surface water and sediment and statistical comparisons of onsite and background metals concentrations are critical to determine whether metals in shoreline media at Iona Island can be attributed to former DoD activities. Con Hook, located approximately 3 miles upstream of Iona Island on the western side of the Hudson River, has similar depositional environments to Iona Island and will be used to represent background surface water and sediment concentrations. Con Hook consists of approximately 7 acres of upland habitat and another 12 acres of a *Phragmites*-dominated tidal marsh (Con Hook Marsh) (Figure I-4). The Active River Subdivision (CSX Transportation) railroad tracks parallels the western boundary of the marsh. Similar to Iona marsh, Con Hook Marsh adjoining the railroad right-of-way may be impacted by contaminants associated with normal operation of the rail line.

11.1.2 Identify the goals of the study; state the decisions to be made to solve the problem

Phase II, including media to be sampled, sampling locations, and analytes to be sampled, has been designed based on the Phase I results. Specific goals for Phase II are as follows:

- Evaluate bioavailability of metals in soil to evaluate potential impacts to terrestrial receptors.

- Confirm whether metals in soil at AOC #16 – Area D (former Dumping Area) related to historical DoD activities are being transported to the Hudson River and Iona Marsh shorelines via the surface water migration pathways identified during Phase I.

- Evaluate whether metals related to historical DoD activities are present in surface water and sediment at concentrations that pose an unacceptable risk to human health and/or ecological receptors.

- Update the site-specific human health and ecological CSMs as data are collected and data gaps are filled.

The following decisions will be made based on results of the Phase II investigation:

- Do soil physical parameters indicate that metals and/or PAHs attributable to DoD activities are bioavailable to ecological receptors?

  — Lines of evidence for bioavailability will be presented in the RSLERA. If physical parameter data indicate that metals and/or PAH concentrations in onsite soil are not bioavailable to ecological receptors, then the RSLERA will conclude that there is no potential significant risk to terrestrial ecological receptors exposed to onsite soil.

  — If physical parameter data indicate that metals and/or PAH concentrations in onsite soil are bioavailable to ecological receptors, **and** if lines of evidence and uncertainty in the RSLERA cannot rule out potential significant risk to ecological receptors, then a Baseline Ecological Risk Assessment (BERA) will be developed to further evaluate potential risks.

- Are metals being transported from AOC #16 – Area D (former Dumping Area) to the shoreline?

  — If constituents detected in Phase II surface water and/or sediment were not detected in Phase I source area soil at AOC #16 – Area D (former Dumping Area), then those constituents in surface water and/or sediment will not be attributed to AOC #16 – Area D (former Dumping Area).

- Are metals from AOC #16 – Area D (former Dumping Area) present in shoreline surface water and/or sediment at concentrations that exceed applicable human health and/or ecological screening values?

— If metals concentrations in surface water and/or sediment are less than or consistent with applicable EPA screening values, then no further action will be recommended for surface water and/or sediment at the Iona Island FUDS.
— If metals concentrations in surface water and/or sediment are greater than applicable EPA screening values, then those analytes exceeding screening values will be further evaluated to determine whether elevated concentrations are a result of DoD activities or a result of Hudson River contamination.

- Are metals concentrations in shoreline media (surface water and/or sediment) consistent with background concentrations at Con Hook? Note, only those metals detected at concentrations greater than applicable EPA screening values will be compared to background data.

  — Metals detected in surface water and/or sediment at concentrations that are less than or consistent with background data from Con Hook (i.e., cannot be contributed solely to AOC #16 – Area D [former Dumping Area] due to Hudson River contamination and/or normal operations of the railroad that parallels the western shoreline of the river) will be excluded from further evaluation in the risk assessments.

  — Metals detected in surface water and/or sediment at concentrations that exceed applicable screening values and background concentrations at Con Hook will be further evaluated in the risk assessments.

- Do the results of the risk assessment indicate unacceptable risk to human receptors exposed to surface water and/or sediment?

  — If results indicate that metals concentrations in surface water and/or sediment may pose potential significant risk to human health, then remedial alternatives will be developed to address those potentially significant risks to move the FUDS forward through the remedial process and determine an appropriate final remedy for the site. It should be noted that only areas with significant risks attributable to DoD activities require remedial action. Only those specific constituents identified as contaminants of concern (COCs) will be carried forward to the Feasibility Study (FS) for development of remedial alternatives.

- Do the results of the RSLERA indicate potential significant risk to ecological receptors exposed to environmental media and the need for a BERA?

  — If the RSLERA indicates that specific metals pose potential significant risk to ecological receptors, **and** if data indicate that those metals are not bioavailable to ecological receptors, then the RSLERA will conclude that there is no potential significant risk to ecological receptors.

— If the RLSERA indicates potential significant risk to ecological receptors, **and** data indicate that those metals are bioavailable to ecological receptors, then additional lines of evidence (such as field observations to determine if there is destruction or compromised habitat) will be further evaluated to further determine potential significant risk.

  - If the RLSERA (including evaluation of bioavailability parameters and additional lines of evidence) concludes that there is no potential significant risk to ecological receptors, then no additional data are needed and a BERA will not be developed.

  - If the RSLERA (including evaluation of bioavailability parameters and additional lines of evidence) concludes that there is potential significant risk to ecological receptors, then a BERA that focuses on the potential significant risk will be developed.

- Do the results of the BERA indicate potential significant risk to ecological receptors exposed to environmental media?

  — If the results of the BERA do not indicate significant risk to ecological receptors, then then no additional data are needed.

  — If the results of the BERA indicate significant risk to ecological receptors, then risk management discussions will explore the need for remedial actions.

11.1.3 Identify the information inputs; identify information and supporting measurements needed to make the decisions and describe the source(s) of the information

Information inputs for Phase II include the following:

- Phase I onsite and offsite/background surface soil (from 0–6 in. bgs) and shallow subsurface soil (from 6–36 in. bgs or refusal) data (Appendix A)

- Information gathered during the Phase I shoreline reconnaissance

- Phase I discrete sediment TOC and grain size analytical data

- Phase I DU location data

- Phase I photogrammetry topographic data collected during the Phase I aerial survey

- Hudson River bathymetry and bedrock surface data (USACE New York District 2018)

- Available historical data from previous studies including the 1996 Data Collection Activities (Greeley-Polhemus 1997) (specifically, PAH concentrations in soil at IX-103-B1, located within DU-13 approximately 50 ft topographically downslope from AOC #1 - Former Building 103 [Paint and Oil Storage])

- Data to be collected during Phase II:

  — Onsite soil physical parameters (TOC, grain size by sieve and hydrometer, CEC, and ORP) to evaluate bioavailability of metals detected in Phase I upland and hydric soil samples

  — Metals (including mercury) data in surface water samples collected within AOC #16 – Area D (Former Dumping Area) during tidal inundation, from the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area), and from Iona marsh at the outlets of the riprap filled drainage swale and tidal wetland swale to evaluate surface water migration pathways from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines

  — Metals (including mercury) surface water and sediment background data from Con Hook to evaluate whether surface water and sediment at Iona Island is impacted from former DoD activities at concentrations that pose significant risk to human health and ecological receptors

  — Surface water hardness data and physical parameter data (i.e., temperature, pH, DO, ORP, TSS, and turbidity) from Iona Island sampling locations established to evaluate surface water migration pathways leading from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines to calculate EPA screening values for aquatic life and support the RSLERA

  — Surface water hardness data and physical parameter data (i.e., temperature, pH, DO, ORP, TSS, and turbidity) from Con Hook to evaluate background representativeness

  — Metals (including mercury) sediment data collected at 0-6 in. bgs from the Iona Island and Con Hook shorelines to evaluate whether sediment is impacted by metals originating from AOC #16 – Area D (former Dumping Area) at concentrations that may pose risk to human health and ecological receptors

  — Sediment physical parameter data (i.e., TOC, grain size by sieve and hydrometer, pH, and ORP) from the Iona Island and Con Hook shorelines to evaluate the bioavailability of metals in sediment and support the development of the RSLERA and to allow comparison of site and background sediment physical characteristics

  — Sediment AVS/SEM data from the Iona Island Hudson River shoreline and Iona Marsh shoreline to assess the bioavailability/toxicity potential to benthic invertebrates.

11.1.4 Define the boundaries of the Study; specify conditions (i.e., time periods and spatial locations)

Phase II sampling is scheduled for late winter / early spring 2022 and includes collection of onsite ISM surface soil samples (0-6 in. bgs), onsite and background discrete surface water samples, onsite and background ISM sediment samples, and onsite discrete AVS/SEM sediment samples (Figures 11-1 through 11-4). Physical boundaries for Phase II include select Phase I DUs (upland and hydric soil); the extent of AOC #16 – Area D (former Dumping Area); the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area); the outlets of the riprap filled drainage swale and tidal wetland swale; and the Con Hook Hudson River and marsh shorelines. Time periods for Phase II sampling are dependent on include tidal fluctuations and precipitation events.

11.1.4.1 Onsite Upland and Hydric Soil Physical Parameter Sampling –ISM

ISM soil sampling will be conducted to evaluate physical parameters (TOC, grain size by sieve and hydrometer, CEC, and ORP) in onsite surface soil (0-6 in. bgs) to qualitatively assess bioavailability of metals and PAHs detected during Phase I and support development of the RSLERA. Grain size results will include gravel, fine/medium/coarse sand, silt, and clay fractions.

Two separate terrestrial environments with different soil types have been identified at Iona Island. These areas include the upland area (upland soil) and the low-lying marsh area between Iona and Round Islands (hydric soil). Based on observations of Phase I soil and field screening of soil pH, physical parameters are not expected to vary significantly within each terrestrial environment. During Phase I, soil types and pH measurements were recorded for 3 ISM samples collected in each onsite DU, and results indicated similar values within each terrestrial environment). Additional soil physical parameters (TOC, CEC, grain size, ORP) are anticipated to have similarly homogenous results.

Surface soil (0-6 in. bgs) will be collected for physical parameters from a total of 8 DUs from across Iona Island. DUs to be sampled in Phase II were selected to provide reasonable spatial coverage/representation across Iona Island. Soil in the upland area was reworked following Navy transfer of the FUDS property to PIPC in 1965, resulting in mixing of surface and subsurface soil throughout and across much of the former operational areas. From a topographic and physical features point-of-view, the physical parameters within the 8 DUs are representative of all conditions at Iona Island and can apply across the island. Therefore, physical parameters from the 8 DUs will be used to conduct a sitewide evaluation of bioavailability parameters, as well as to conduct an evaluation on a DU-by-DU basis.

Five separate upland soil DUs were selected to represent the upland area including DU-4, DU-7, DU-9, DU-11, and DU-13. Phase II parameters from these DUs are expected to be representative of upland soil conditions across Iona Island. Additionally, hydric soil will be collected from each of the three Phase I DUs within the marsh area between Round and Iona Island where elevated concentrations of metals and/or PAHs were reported during Phase I (DU-17, DU-18, and DU-19).

Phase II parameters from these DUs are expected to be representative of hydric soil conditions across AOC #16 – Area D (former Dumping Area).

Phase II soil physical parameter data will be collected using ISM, which is consistent with Phase I soil sampling and will allow for obtaining better estimates of mean values of parameters of interest across DUs. Samples at each onsite DU will be collected in triplicate (initial ISM sample plus two additional samples) to allow calculation of the Chebyshev 95% upper confidence limit of the mean (95UCL) at each DU, which is consistent with Phase I onsite soil sampling. The Chebyshev 95UCL for each DU will be calculated using the ITRC calculator (ITRC 2020). In addition, a 95UCL will be generated for the entirety of Iona Island to support evaluation of bioavailability in DUs that are not being sampled in Phase II.

Onsite DUs evaluated during Phase II are sized at approximately 1-acre each (200 ft x 200 ft). Each Phase II ISM soil physical parameter sample will be comprised of 50 increments of soil to estimate the reasonable unbiased estimate of concentrations across each DU, which is consistent with Phase I soil sampling. Surface soil increments will be collected at a depth of 0–6 in. bgs using plungers capable of collecting sediment equivalent to approximately 33 grams (g) in volume, to produce a single sediment sample equivalent to approximately 1 kg.

11.1.4.2 Surface Water Metals Sampling – Discrete

Surface water sampling will be conducted to evaluate whether metals are being transported from AOC #16 – Area D (former Dumping Area) to the shoreline environment at concentrations that may pose risk to human health and/or ecological receptors. Onsite areas targeted for investigation during Phase II to evaluate the contaminant migration pathway include the source area at AOC #16 – Area D (former Dumping Area) and surface water migration pathway endpoints (Hudson River along the eastern boundary of AOC #16 – Area D [former Dumping Area] and Iona Marsh at the outlets of the riprap filled drainage swale and tidal wetland swale) (Figure 11-2a). For discrete sampling, sample sizes of at least 20 are typically needed to compare mean site concentrations with mean background concentrations using hypothesis tests for centrality. Therefore, 20 sample locations will be collected from each onsite area targeted for investigation during Phase II (source area, Hudson River shoreline, and Iona Marsh shoreline) to ensure the sample sizes for discrete samples are adequate for normality tests.

Spatial areas and time periods for discrete surface water sampling include the following:

- Twenty discrete surface water samples collected throughout AOC #16 – Area D (former Dumping Area) to evaluate whether contaminants are being mobilized from soil within the source area to tidal water (DUMP-SW-01 through DUMP-SW-20).

- Twenty discrete surface water samples from the Hudson River along the eastern boundary of AOC #16 – Area D (former Dumping Area), with surface water collected during high or receding tide to evaluate potential migration of contaminants from AOC #16 – Area D (former Dumping Area) to the river shoreline (IHRS-SW-01 through IHRS-SW-20)

- Twenty discrete surface water samples along the Iona Marsh shoreline to evaluate potential migration of contaminants from AOC #16 – Area D (former Dumping Area) to the marsh shoreline. Sampling locations will be concentrated at the outlets of the riprap filled drainage swale (RRDS-SW-01 through RRDS-SW-10) and tidal wetland swale (TWS-SW-01 through TWS-SW-10) to further evaluate these swales as contaminant migration pathways.

Background surface water sampling will be conducted at Con Hook upstream from Iona Island to determine regional background and/or upstream concentrations of TAL metals (including mercury) and support development of risk assessments. Discrete surface water samples will be collected from both the Hudson River and marsh sides of the island at low tide to establish regional background/upstream metals concentrations for each depositional environment as follows (Figure 11-3). A total of 20 samples will be collected from each depositional environment to ensure the background sample sizes are adequate for normality tests.

- Twenty discrete background surface water samples along the Con Hook Hudson River shoreline (CHHRS-SW-01 through CHHRS-SW-20), with surface water collected during low tide to evaluate background concentrations in Hudson River water.

- Twenty discrete background surface water samples along the Con Hook Marsh shoreline, (CHMS-SW-01 through CHMS-SW-20), with surface water collected during low tide to evaluate background concentrations in marsh water.

11.1.4.3 Shoreline Sediment Metals Sampling – ISM

Sediment sampling will be conducted to determine if metals associated with AOC #16 – Area D (former Dumping Area) have been transported to the Hudson River and Iona Marsh shorelines and accumulated in shoreline sediment at concentrations that may pose risk to human health and/or ecological receptors. Phase II sediment samples for metals analyses will be collected using ISM to allow for obtaining better estimates of DU means, which is consistent with surface soil sampling conducted during Phase I.

Three sediment DUs, each sized at approximately ½-acre, have been established for conducting sediment sampling at Iona Island, with one DU established along the Hudson River shoreline and two DUs established along the Iona Marsh shoreline. DUs were located at the ends of preferential flow pathways from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines. The approximately ½-acre size of these DUs was established as this represents the smallest area for which ecological receptors may be exposed. The muskrat, which will represent herbivorous aquatic mammals, has an approximately ½-acre foraging range. DUs are presented in Figure 11-2b. Spatial areas and time periods for sampling include the following:

- One DU along the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area), with ISM sampling during low tide when shoreline sediment is

exposed to evaluate whether metals have been transported to the Hudson River shoreline and accumulated in shoreline sediment (IHRS-DU-01)

- One DU along Iona Marsh shoreline at outlet of the riprap filled drainage swale, with ISM sampling during low tide when shoreline sediment is exposed to evaluate whether CERCLA hazardous metals from AOC #16 – Area D (former Dumping Area) been transported via the swale to the marsh shoreline and accumulated in sediment (RRDS-DU-01)

- One DU along Iona Marsh shoreline at outlet of the tidal wetland swale, with ISM sampling during low tide when shoreline sediment is exposed to evaluate whether metals from AOC #16 – Area D (former Dumping Area) been transported via the swale to the marsh shoreline and accumulated in sediment (TWS-DU-01).

The Chebyshev 95UCL will be used to represent exposure point concentrations (EPCs) for sediment, similar to that done during Phase I. The Chebyshev 95UCL is a more conservative estimate of the UCL than the Student t-Test 95UCL because it achieves or exceeds the desired coverage rates, even for non-normally distributed data. The Chebyshev 95UCL will be calculated using the ITRC calculator (ITRC 2020) and directly compared with appropriate risk-based screening values. It takes a minimum of three replicates to calculate the 95UCL using statistical tests. Therefore, ISM sediment samples at each onsite DU will be collected in triplicate (initial ISM sample plus two additional samples).

Background sediment sampling will be conducted upstream from Iona Island to evaluate regional background and/or upstream concentrations of metals (including mercury). Sampling will be conducted using ISM during low tide, consistent with Iona Island shoreline sampling. Two DUs have been established at Con Hook, with one DU on the Hudson River shoreline (CHHRS) and one DU on the marsh shoreline (CHMS), to evaluate background concentrations in each depositional environment. Due to the special characteristics associated with incremental sampling (low variance, small sample sizes), and because of the complex history of the Hudson River and potential upstream sources, background comparison requires careful consideration. For this project, each background DU consists of eight non-contiguous SUs, consistent with the Phase I soil background investigation. Due to spatial constraints along the shoreline at Con Hook, each SU was sized at 0.15 acres (162 x 40.5 ft along the Hudson River shoreline and 81 ft x 81 ft along the marsh shoreline). Single samples will be collected from each SU, consistent with the Phase I soil background investigation, to provide a background dataset of 8 samples for each depositional environment as follows:

- One ISM sample will be collected from each of 8 SUs along the Hudson River shoreline of Con Hook, with ISM sampling during low tide when shoreline sediment is exposed to evaluate background concentrations of metals (including mercury) (CHHRS-SU-01 through CHHRS-SU-08)

- One ISM sample will be collected from each of 8 SUs along the Con Hook Marsh shoreline, with ISM sampling during low tide when shoreline sediment is exposed to evaluate background concentrations of metals (including mercury) (CHMS-SU-01 through CHMS-SU-08).

Increments for each Iona Island and Con Hook background sediment ISM sample will be collected at a depth of 0–6 in. bgs, which is representative of the biological active zone. EPA SW846 Method 8330B recommends collecting 30 or more evenly spaced increments to build a sample with a total mass greater than 1 kilogram (kg). Therefore, each ISM sediment sample will consist of 50 increments of sediment, which is sufficient to estimate the reasonable unbiased estimate of concentrations across each onsite DU and each background SU. Sediment increments will be collected using plungers capable of collecting sediment equivalent to approximately 33 grams (g) in volume, to produce a single sediment sample equivalent to approximately 1 kg.

Due to the orientation of Iona Island ½-acre sized sediment DUs and Con Hook background ½-acre sized SUs along the Hudson River, Iona Marsh, and Con Hook Marsh shorelines, and the variability of the shoreline environment (i.e., presence/absence of sediment, surface water depth, exposed bedrock, etc.), locations for each sediment sample increment will be determined using the simple random sampling approach (ITRC 2020). Increments in each onsite DU and each background SU will not be collected from the same location or co-located together.

11.1.4.4 Shoreline Sediment Physical Parameter Sampling – ISM

Sediment physical parameter data (grain size by sieve and hydrometer, TOC, pH, and ORP) provide information on bioavailability of metals to indigenous benthic organisms. Therefore, sediment sampling will be conducted during Phase II at each onsite sediment DU to evaluate bioavailability and support development of the RLSERA. Grain size results will include gravel, fine/medium/coarse sand, silt, and clay fractions.

Sediment physical parameter data (TOC, grain size by sieve and hydrometer, pH, and ORP) will be collected from 0-6 in. bgs at each onsite sediment DU using ISM, with samples collected in triplicate (initial ISM sample plus two additional samples) to obtain better estimates of DU means (Figure 11-2b). Background physical parameter ISM samples will be collected at each Con Hook background ISM sediment SU to evaluate background representativeness, providing a total of 8 samples for each background DU (each depositional environment) (Figure 11-3). ISM sediment physical parameter samples will be collected concurrent with ISM sediment metals samples.

11.1.4.5 Shoreline Sediment AVS/SEM Sampling - Discrete

AVS/SEM data provides information on the potential for metal ions found in sediment to cause toxic effects in organisms dwelling in the sediment. The AVS/SEM analytical method is predicated on maintaining anoxic conditions required to sustain metal sulfide concentrations. Exposure of sediment samples to oxygen during sample collection and analysis can result in false positive assertions of metal bioavailability. Samples must be placed in sample jars with zero

headspace immediately upon collection. These samples cannot be collected using ISM, which will expose sediment to oxygen during collection. Therefore, AVS/SEM samples will be collected as discrete grab samples, rather than ISM samples. As background sediment toxicity is irrelevant to onsite sediment toxicity, AVS/SEM samples will only be collected from onsite locations.

Three discrete AVS/SEM sediment samples will be collected in each onsite sediment DU to evaluate the bioavailability potential of 5 divalent metals (cadmium, copper, nickel, lead, and zinc) in the RSLERA. Samples will be collected at depths of 0-6 in. bgs using grab sampling methods. Sample locations are presented on Figure 11-2b.

- 3 discrete samples collected from the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area) (i.e., within IHRS-DU-01) (IHRS-SED-01 to IHRS-SED-03)

- 3 discrete samples collected from the Iona Marsh shoreline at the outlet of the riprap filled drainage swale (i.e., within RRDS-DU-01) (RRDS-SED-01 to RRDS-SED-03)

- 3 discrete samples collected from the Iona Marsh shoreline at the outlet of the tidal wetland swale (i.e., within TWS-DU-01) (TWS-SED-01 to TWS-SED-03).

Although AVS/SEM data will not be quantitatively evaluated by statistical methods, the data allow a qualitative conclusion to be made about the potential for bioavailability of 5 divalent metals in sediment. If the RSLERA proceeds to a BERA, the AVS/SEM results may assist in limiting the effort (e.g., toxicity testing) required as part of the BERA, by focusing on a specific area or metal that could be driving risk to benthic invertebrates.

11.1.5 Develop the analytic approach

Prior to conducting Phase II field sampling, the eQAPP developed within Formerly Used Defense Site Online Chemical Database (FUDSChem) for Phase I will be updated to include Phase II analytes. The eQAPP will be fully compliant with the project UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum, incorporating methods, sample types, and quality specifications (limit of detection [LOD], limit of quantitation [LOQ], Holding Time, QC Sample Acceptance Limits, etc.).

11.1.5.1 Onsite Upland and Hydric Soil Physical Parameter Sampling – ISM

Onsite soil physical parameter data will be used to evaluate bioavailability of metals detected in upland and hydric surface soil (0–6 in. bgs) during Phase I. ISM soil samples will be submitted for offsite laboratory analysis and onsite field screening as follows:

- TOC by Walkley-Black
- Grain size by ASTM D422 (sieve and hydrometer)
- CEC by EPA SW-846 Method 9081
- ORP by field screening (field and standard hydrogen electrode corrected values).

ISM sample preparation will be completed by Eurofins TestAmerica Canton, OH. ISM soil samples will be air dried, disaggregated, and sub-sampled to create aliquots for TOC, grain size, and CEC. Aliquots for grain size and CEC will not be ground/milled, as grinding would change sample grain size and adsorption capacity. Aliquots for TOC analysis will be ground and sieved per the analytical method. Grain size and TOC aliquots generated from sample preparation will be sent to Katahdin Analytical Services, LLC, Scarborough, ME for analysis. CEC aliquots will be sent to Eurofins TestAmerica, Corpus Christi, TX for analysis. Analytical results will be presented in the RI report. Bioavailability of metals and PAHs to ecological receptors will be qualitatively evaluated in the ERA.

11.1.5.2 Surface Water Metals Sampling – Discrete

Iona Island surface water metals (including mercury) data are needed along surface water migrations leading from AOC#16– Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines to evaluate the potential migration of metals contaminants from AOC #16 – Area D (former Dumping Area) to the shorelines. Background surface water data at Con Hook are needed to evaluate whether constituents detected in Iona Island samples can be attributed to former DoD activities at AOC#16– Area D (former Dumping Area). As EPA screening values for metals are hardness dependent, hardness data are needed for surface water sampling locations. Discrete surface water samples will be submitted for offsite laboratory analysis of the following:

- Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A
- Dissolved (field filtered) mercury by EPA Method 7470A
- Hardness by SM2340.

For completeness purposes, the full list of TAL metals is being evaluated for surface water samples. However, those metals that are not a CERCLA hazardous substance (for example, manganese) will be ineligible for DERP FUDS response.

Laboratory analytical services for surface water samples will be provided by Eurofins TestAmerica Denver, CO. In addition, surface water at each sampling location will be screened in the field for water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) for the purpose of comparing background representativeness, and collecting data to support the risk assessments.

Surface water TAL metals (including mercury) results will be compared to applicable EPA screening values in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as follows. Analytes without screening values will be reported for consideration in the risk assessments. If analytes without established risk-based screening values are detected, these analytes will be assessed using alternative means such as use of surrogate compounds.

- Human health screening values: EPA Safe Drinking Water Act maximum contaminant levels (MCLs) and tapwater RSLs (most recent version at the time of report preparation). The lesser of the two values will be prioritized for use in the HHRA.

- Ecological screening values: The default surface water screening values were selected from currently available National Recommended Water Quality Criteria (NRWQC) for aquatic life, chronic freshwater (available: https://www.epa.gov/wqc/national-recommended-water-quality-criteria-aquatic-life-criteria-table). For analytes without currently available NRWQC, additional values were selected from EPA compilations. Due to a lack of compiled EPA surface water screening values for Region 2 (the State of New York's EPA region), screening values from EPA Regions 3 and 4 were compared and are presented in Table 11-1. These values include compilations from outdated NRWQC, the Great Lakes Initiative (GLI 2013), Suter et al. (1996), and the Canadian Council of Ministries of the Environment (CCME 2003). The hierarchy for selection of screening values was: NRWQC (current values), GLI (2013), and Suter et al. (1996). CCME values were not selected as these are Canadian numbers. Bold values indicate the selected screening values for each analyte, which are presented along with laboratory limits in Worksheet #15.

Source area/upstream and shoreline TAL metals (including mercury) constituents will be evaluated to determine if metals concentrations in shoreline surface water are a result of AOC #16 – Area D (former Dumping Area). Constituents detected in shoreline surface water that were not detected in onsite source area/upstream media (soil and surface water) will not be attributed to AOC #16 – Area D (former Dumping Area).

Background concentrations obtained from Con Hook will be used to determine if onsite shoreline concentrations are consistent with or less than background concentrations. A quantitative statistical approach (statistical hypothesis tests) will be used to compare onsite and background surface water results. Samples collected along the Hudson River shoreline at Iona Island will be compared to background samples collected along the Hudson River shoreline at Con Hook. Samples collected along the Iona Marsh shoreline will be compared to background samples collected along the Con Hook Marsh shoreline. Concentrations that exceed both applicable EPA screening values and background concentrations will be further evaluated in the HHRA and/or ERA.

11.1.5.3 Shoreline Sediment Sampling – ISM

Iona Island and Con Hook background sediment metals (including mercury) data are needed to evaluate whether metals contaminants from AOC #16 – Area D (former Dumping Area) have accumulated in shoreline sediment at concentrations that may pose risk to human health and/or ecological receptors. In addition, if metals concentrations exceed both background concentrations and applicable ecological screening values, then potential bioavailability of metals to ecological receptors will be evaluated in the ERA. Therefore, Iona Island sediment physical parameter data is needed to evaluate bioavailability of metals from AOC #16 – Area D (former Dumping Area)

in sediment and support the ERA. In addition, Con Hook sediment physical parameter data is needed to evaluate background representativeness.

ISM sediment samples will be submitted for offsite laboratory analysis and onsite field screening as follows:

- Metals by EPA Methods 6010C/6020A
- Mercury by EPA Method 71471B
- TOC by Walkley-Black
- Grain size by ASTM D422 (sieve and hydrometer)
- pH by field screening
- ORP by field screening (field and standard hydrogen electrode corrected values).

Sediment samples will be analyzed for TAL metals. While nutrient metals (calcium, magnesium, potassium, and sodium) are included in the analyte list to help evaluate sediment geochemistry and ultimately bioavailability, no PALs were identified for screening of data for these metals in sediment and therefore they are not included in Worksheet #15. Additionally, nutrient metals are not CERCLA hazardous substances and are ineligible for DERP FUDS response.

ISM sample preparation will be completed by Eurofins TestAmerica Canton, OH. Each ISM sediment sample will be air dried and disaggregated. Each sample will then be subsampled for a grain size aliquot, and the remainder will be sieved and ground using the ball mill and then subsampled to create aliquots for metals/mercury and TOC analysis. Grain size and TOC aliquots generated from sample preparation will be sent to Katahdin Analytical Services, LLC, Scarborough, ME for analysis. Aliquots for metals analysis will be sent to Eurofins TestAmerica Denver for analysis. Analytical data will be presented in the RI report.

Ball mill grinding will be utilized during laboratory sample processing of metals/mercury and TOC samples, consistent with Phase I. As was done during Phase I, grinding will be conducted using a rolling ball mill with steel paint cans and ceramic cylinders (90% burundum) as grinding media. Milling durations will be consistent between samples, as conducted for Phase I samples and consistent with ITRC guidance (ITRC 2020). It is noted that ball milling may be associated with cross-contamination of sediment samples with aluminum and iron, which are not COPCs at the site but may be used in geochemical evaluation of the sediments. The laboratory will perform grinding blanks using well-characterized sand to allow assessment of the degree of cross-contamination with these metals.

Metals concentrations will be compared to applicable EPA human health and ecological risk-based screening values in accordance with CERCLA. Risk-based EPA screening values that are protective of human health and ecological risks are presented in Worksheet #15 of this UFP-QAPP Addendum. Consideration will be given to other screening values through the CERCLA applicable, or relevant and appropriate requirements (ARAR) process with the input of Counsel. Analytes without screening values will be reported for consideration in the risk assessments. If

analytes without established risk-based screening values are detected, these analytes will be assessed using alternative means such as use of surrogate compounds.

- Human health screening values: EPA residential and industrial RSLs for soil (most recent version at the time of report preparation). Note: Industrial RSLs will be used for comparison purposes only. For the HHRA, residential soil RSL (with hazard quotient [HQ] adjusted from 1 to 0.1[6]) will be used to determine COPCs in onsite sediment. A deed restriction is in place noting that there is to be no residential development at the site (USACE 1997); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

- Ecological screening values: EPA Region 4 (March 2018) ecological screening values for sediment. Due to a lack of EPA sediment screening values for Region 2 (the State of New York's EPA region), EPA's most recent (i.e., 2018) compilation of sediment screening values for Region 4 will be used, since values are the same (or lower) than those presented by Region 3. Freshwater sediment screening values (Ecological Screening Values [ESV]) will be compared to sediment data for the SLERA. Less conservative Revised Screening Values (RSV) will be incorporated into the RSLERA. Values are presented in Appendix G and in Worksheet #15, along with laboratory limits. These values include the following sources of peer-reviewed, scientific literature:

    - Long et al. 1991. The Potential for Biological Effects of Sediment-Sorbed Contaminants Tested in the National Status and Trends Program. NOAA Technical Memorandum NOS OMA 52. Effects range low (ER-L) for chronic and effects range medium (ER-M) for acute.

    - MacDonald et al. 2003. Development and Evaluation of Numerical Sediment Quality Assessment Guidelines for Florida Inland Waters. Florida Department of Environmental Protection, Tallahassee, FL. Development and Evaluation of Numerical Sediment Quality Assessment Guidelines for Florida Inland Waters. Threshold effect concentration (TEC) for the ESV and probable effect concentration (PEC) for the RSV.

    - MacDonald, D.D. 1994. Approach to the Assessment of Sediment Quality in Florida Coastal Waters. Florida Department of Environmental Protection. 1994 Florida Sediment Quality Assessment Guidelines for Florida Coastal Waters.

    - Persaud et al. 1993. Guidelines for the protection and management of aquatic sediment quality in Ontario. Ontario Ministry of the Environment. Queen's Printer of Ontario.

[6] For non-carcinogens, the RSLs are based on a HQ of 1.0. To account for potential cumulative effects of multiple contaminants affecting the same target organ, one-tenth of the acceptable non-carcinogenic threshold was used for screening.

- Washington State Sediment Management Standards, Cleanup Objectives

- Los Alamos National Laboratory ECORISK Database. September 2017. http://www.lanl.gov/environment/protection/eco-risk-assessment.php.

Background concentrations in sediment samples obtained from Con Hook will be used to determine if concentrations in Iona Island sediment are consistent with or less than background concentrations. Evaluations will be conducted for each sediment depositional environment (Hudson River and marsh shoreline). Samples collected along the Hudson River shoreline at Iona will be compared to background samples collected along the Hudson River shoreline at Con Hook. Samples collected along the Iona Marsh shoreline will be compared to background samples collected along the Con Hook Marsh shoreline.

Sufficient background sediment samples will be collected for each depositional environment so that statistics such as Upper Predictive Limits (UPL) will be calculated. To minimize false positives (i.e., erroneously concluding site concentration is elevated relative to background concentrations), site concentrations will be compared with UPLs for the next k site observations. (In general, the background UPL calculation depends on factors that include the level of confidence–[1 - α], the background sample size [n], and the number of future site observations [k].)

Permutation test of means (Student t-Test with adjusted p-value) was used during Phase I to evaluate whether onsite metals and PAH concentrations in terrestrial soil exceeded regional background metals and PAH concentration ranges. Therefore, based on the Phase II results (i.e., if the distribution of site data is significantly higher than the distribution of background data and if the data are nonparametrically distributed), then permutation test of means will be used during Phase II to be consistent with Phase I. Information on permutation testing is presented in Worksheets #14 and #16 of this UFP-QAPP Addendum.

CERCLA hazardous metals concentrations that exceed both background concentrations and applicable EPA screening values will be further evaluated in the HHRA and/or ERA.

Bioavailability of metals to ecological receptors will be qualitatively evaluated in the RSLERA. In addition, Iona Island and Con Hook sediment physical parameter data will be compared to evaluate whether background sampling locations are consistent with onsite locations. Evaluations will be conducted for each depositional environment. Samples collected along the Hudson River shoreline at Iona Island will be compared to background samples collected along the Hudson River shoreline at Con Hook. Samples collected along the Iona Marsh shoreline will be compared to background samples collected along the Con Hook Marsh shoreline. Permutation test of means (Student t-Test with adjusted p-value) will be used to compare the site physical parameters with the background physical parameters.

11.1.5.4 Shoreline Sediment AVS/SEM Sampling - Discrete

AVS/SEM data are needed to assess the bioavailability/toxicity potential of metals to benthic invertebrates. Discrete sediment samples will be submitted for offsite laboratory analysis of AVS/SEM by SM 4500-S2D and EPA Method 6010C. Laboratory analytical services for AVS/SEM will be provided by Eurofins Lancaster Laboratories, Inc., PA.

11.1.6 Specify Performance or Acceptance Criteria

This UFP-QAPP Addendum, in conjunction with the UFP-QAPP (EA 2020a), provides a description of the data analysis and QA/QC measurements to be conducted during Phase II. The QA/QC measures are used to evaluate and document the quality of the data that are obtained, which will reduce the likelihood that the planned approach could result in a failure to achieve the project objectives.

The data need to be of adequate quality to make the decisions established for the site. The purpose of this is to minimize the possibility of either making erroneous conclusions or failing to keep uncertainty in estimates to within acceptable levels. Worksheet #12 and Worksheet #28 of this UFP-QAPP Addendum, in conjunction with Worksheet #12 of the UFP-QAPP (EA 2020a), presents the analytical measurement performance criteria for surface water, sediment, and soil samples applicable to the analytical sampling associated with Phase II.

Laboratory analytical services for TAL metals by EPA Methods 6010C/6020A, mercury by EPA Method 7470/7471B, AVS/SEM by SM 4500-S2D and EPA Method 6010C, TOC by Walkley-Black, and grain size by ASTM D422 will be provided by DoD-certified laboratories. Laboratory certifications are provided in Appendix C of this UFP-QAPP Addendum. Laboratory analyses will be conducted in accordance with the DoD QSM Version 5.3 (2019). Laboratory detection limits (MDLs, LODs, and LOQs) are provided in Worksheet #15, alongside the project action limits (PALs). As part of the data review, analytes with LOQs and LODs that are higher than screening values will be discussed by the PDT once the analytical data are received and prior to the risk evaluation. In addition, any data that are reported to the LOQ and J-qualified will also be reviewed for potential quality issues, trends, and/or historical use.

Note that Eurofins TestAmerica, Canton is not DoD ELAP accredited for ISM processing via ball milling. EA was unable to identify a DoD ELAP accredited laboratory to perform the ISM processing with ball milling. Therefore, following review of the laboratory SOP, the USACE Project Chemist agreed to allow Eurofins TestAmerica in Canton, OH to process ISM sediment samples. DoD has not accredited any laboratories for CEC by EPA SW-846 Method 9081, however the USACE Project Chemist has agreed to have Eurofins TestAmerica Corpus Christi, TX perform CEC analysis. Although CEC is not included in the DoD QSM, Version 5.3 (DoD 2019), QA/QC requirements per the QSM will be followed to the extent possible. Eurofins TestAmerica in Denver, CO does not have DoD ELAP accreditation for hardness by Method 2340B, however Eurofins TestAmerica Denver, CO does have National ELAP accreditation for this method. In addition, hardness by Method 2340B is a calculation method based on calcium

and magnesium concentrations from EPA Method 6010C. Eurofins TestAmerica Denver, CO does have DoD ELAP accreditation for EPA Method 6010C, and the USACE Project chemist has agreed to have Eurofins TestAmerica Denver, CO perform the hardness calculation.

The quality of sampling and analysis must be at a level that results in representative and reproducible data. The data generated must be sufficiently precise and accurate to meet project objectives and be appropriate for the intended use. Data of sufficient quality for use in evaluation and decision making in this project will be defined as data that are produced following the specified SOPs and meeting the established criteria in this UFP-QAPP Addendum, in conjunction with the UFP-QAPP (EA 2020a), including precision, accuracy, comparability, representativeness, completeness, and sensitivity. Analytical data will be validated as described in Worksheet #34 and #35 of the UFP-QAPP (EA 2020a) and Worksheet #36 of this UFP-QAPP Addendum. Data usability will be assessed as described in Worksheet #37 UFP-QAPP (EA 2020a). A discussion of acceptable limits for decision errors related to data screening is presented under the heading "How good do the data need to be to support the environmental decision," below.

Iona Island surface water and sediment analytical data will be compared to appropriate EPA screening values to determine if site concentrations exceed human health and ecological risk screening values. Discrete water data will be directly compared to appropriate risk screening values presented in Worksheet #15 of this UFP-QAPP Addendum. Sediment DUs at Iona Island will be sampled in triplicate and the Chebyshev 95UCL will be calculated using the calculator included with ITRC (2020). The Chebyshev 95UCL will be directly compared with appropriate risk screening values risk screening values for sediment presented in Worksheet #15 of this UFP-QAPP Addendum. The use of the Chebyshev 95UCL for this decision is consistent with ITRC (2020) guidance and Phase I investigation.

Background surface water and sediment metals analytical data generated from sampling at Con Hook will be used to determine if metals concentrations in surface water and sediment at Iona Island are consistent with or exceed naturally occurring and/or upstream metals concentrations. In addition, Iona Island discrete surface water and ISM sediment physical parameter data will be compared to background discrete surface water and ISM sediment data to ensure background sampling locations are representative of onsite conditions. Onsite and background surface water physical parameters will be compared using a quantitative statistical approach (statistical hypothesis tests). Onsite and background ISM sediment physical parameter data will be compared using permutation test of means at the 95% confidence level as presented in Worksheet #14 and #16.

11.1.7 Develop the plan for obtaining data; evaluate the results of the previous steps and develop the most resource-efficient design for data collection

The plan for data collection is as follows:

- ISM surface soil physical parameter samples will be comprised of 50 random increments from onsite approximately 1-acre sized (200x200 ft) DUs. Increments will be collected

from 0–6 in. bgs using a dedicated incremental sampling tool with plungers collecting soil equivalent to approximately 33.3 g in volume, which will provide a total weight of 1 kg of sediment per sample.

- Discrete surface water samples will be collected directly into laboratory-provided bottles or with a dedicated (disposable) dipper cup, clean/decontaminated dipper with stainless-steel sampler, or pond sampler.

- ISM sediment samples will be comprised of 50 random increments from onsite ½ acre DUs and background 0.15-acre SUs, each collected from 0–6 in. bgs using a dedicated incremental sampling tool with plungers collecting soil equivalent to approximately 33.3 g in volume, which will provide a total weight of 1 kg of sediment per sample.

- Discrete sediment AVS/SEM samples will be collected from 0–6 in. bgs using dedicated sampling equipment (disposable scoops/spoons) and/or clean/decontaminated, non-dedicated sampling equipment (petite ponar grab, hand augers, or shovels).

- Laboratory analytical samples will be placed into sample collection bags (ISM soil and sediment samples) or pre-preserved bottles (discrete surface water samples, and discrete sediment AVS/SEM samples) provided by the analytical laboratory and shipped offsite for analysis of parameters presented in DQO Step #5.

- Field screening of water quality parameters (surface water samples), ORP (ISM surface soil and sediment samples), and pH (ISM sediment samples) will be conducted in the field using calibrated water quality meters.

- Field observations and field screening data will be in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

Additional details pertaining to the sampling plan are provided in subsequent worksheets as follows:

- Worksheet #14 and #16 in this UFP-QAPP Addendum provides the Phase II project tasks and schedule.

- Worksheet #17 in this UFP-QAPP Addendum provides the Phase II sample design and rationale.

- Worksheet #20 in this UFP-QAPP Addendum provides information on Phase II QC samples and details the field sample count.

- Worksheets #24 and #28 in the UFP-QAPP (EA 2020a) and in this UFP- QAPP Addendum provide specific detail on the Phase II analytical requirements.

- Worksheet #26 and #27 in this UFP-QAPP Addendum, supplemented by the UFP-QAPP (EA 2020a) provides information on sample nomenclature, packaging, shipment, and delivery to the analytical laboratory.

11.2 WHO WILL USE THE DATA?

The RI data will primarily be used by EA and USACE in conjunction with New York State Department of Environmental Conservation (NYSDEC) and PIPC to further characterize the Iona Island Naval Ammunition Depot FUDS and for decision making on the path forward. Data will be reviewed and used by the following:

Analytical data will be validated by the EA Project Chemist and Data Manager. Data validation reports will be uploaded to the FUDSChem library for approval by the USACE chemist. A data usability assessment will be prepared by the EA Project Chemist and Data Manager and presented in the RI report. Validated analytical data will be reviewed by the EA Project Geologist, summarized in analytical tables and figures, and presented in the RI report. The EA Human Health and Ecological Risk Assessors will use validated and usable analytical data to complete the HHRA and ERA.

The RI report and risk assessments will be submitted to USACE for review by the USACE PM, USACE TM, USACE Project Geologist, USACE Project Chemist, USACE Human Health Risk Assessor, and USACE Ecological Risk Assessors. Following approval by USACE, the RI report and risk assessments will be submitted to the USACE Center of Expertise for review and approval. Following approval by the USACE Center of Expertise, the RI report and risk assessments will be submitted to the NYSDEC PM and PIPC Science Director for review and approval.

11.3 WHAT WILL THE DATA BE USED FOR?

The Phase II data will be used to close data gaps identified following completion of Phase I, as presented in Worksheet #10 of this UFP-QAPP Addendum.

11.3.1 Onsite Soil Physical Parameter Data

Onsite soil physical parameter data will be used to qualitatively evaluate bioavailability of COPCs detected in soil and support the RSLERA. Grain size, TOC, ORP, and pH data will be used as screening tools to make estimates of solubility and contaminant availability. CEC in soil will be used in conjunction with grain size and TOC content to qualitatively evaluate the potential bioavailability of metals in soil, particularly for uptake to plants and earthworms. Analytical data will be used to evaluate bioavailability potential for each soil and sediment DU sampled. Onsite soil data will also be used to conduct a site-wide evaluation of soil bioavailability potential.

Physical parameters will be reviewed in conjunction with the COPC analytical data by a geochemist. There are no definitive quantitative ranges that can be used for all COPCs. Each

COPC that exceeds screening values will be evaluated in conjunction with the geochemical parameters to evaluate bioavailability potential.

11.3.1.1 Grain Size

Grain size provides an indication of the grain or particle surface area and can be used to estimate soil permeability and to better understand chemical release from the solid. Particle size provides screening information on the rate and magnitude of contaminant sorption/desorption, which can control bioavailability. Smaller particles have larger surface area to mass ratios and tend to demonstrate higher levels of solubility for metals, thereby decreasing their bioavailability in soil.

In sediment, very small clay particles (up to 0.004 mm) exhibit a strong influence from electrical charges on their surface, resulting in cohesive forces. Particle sizes between 0.004 mm and approximately 0.0625 mm are known as silt and are in a transition range. The silt particles are too large to feel much influence from the electromotive forces and too small to mobilize inertia against flowing water. When particle size exceeds 0.0625 mm, electromotive forces are insignificant. These particles are non-cohesive and are classified as sand, gravel, cobble, etc. (Thomas 1977).

Power and Chapman (1992) proposed that sediments can generally be classified into two groups: coarse, with a grain size > 62 microns (µm), or fine, with a grain size < 62 µm. The coarse fraction is composed primarily of stable, inorganic silicate materials that are non-cohesive and generally not associated with chemical contamination. The fine fraction consists of particles with a relatively large surface area to volume ratio. Typically, surface electric charges cause the fine particles to be more chemically and biologically active, thereby increasing the likelihood of sorption and desorption of contaminants.

11.3.1.2 Total Organic Carbon

TOC is a measure of the organic carbon contained within soil. Metal cations sorb to soil organic matter, and strong adsorption to organic matter reduces the solubility of metals in soil. Increased organic content (TOC) is an indicator of potentially reduced bioavailability. Organic carbon content can be used to obtain a reasonably accurate estimate of the sorption distribution coefficient for hydrophobic organic compounds in soil or sediment samples that have ≥ 0.5 percent carbon (wt/wt) (Allen-King et al. 2002).

11.3.1.3 pH and Oxidation Reduction Potential

Soil pH and soil ORP influence the stability of existing metal phases, weathering, and incorporation of metals into mineral phases, as well as the degree and strength of adsorption of metals to soil. In general, metal mineral dissolution and solubility increase as soil pH decreases (i.e., in acidic soil), and metal solubilities increase as redox potential decreases.

In general, acidic soils with pH less than 6 will have more bioavailable metals than neutral or alkaline soils. Bioavailability from moist or water saturated soils and sediments goes up in general

with lower pH because most metals become more soluble at lower pH. In general soil and sediment moisture pH values lower than 5 to 6 will result in greater metal solubility and bioavailability. Metal concentrations and bioavailability generally increase by an order of magnitude with each pH unit drop from neutrality. Above these pH levels most metals will precipitate as hydroxides and metal solubility and bioavailability decreases dramatically. However, oxyanion species of metals like arsenic and antimony have negative charge and become more soluble at higher than neutral pH.

Metal speciation is a key factor in bioavailability and ligands like dissolved organic compounds or anions like carbonates, sulfate and sulfide may render metals less bioavailable than free metal ions by decreasing thermodynamic activity. In general, high TDS in the range of 100 to 500 mg/l or ionic strengths of 0.01 can be protective especially if the alkalinity is high, higher than 100 mg/l equivalent calcium carbonate. Hardness can also lower bioavailability by competition with tissue uptake sites. Increasing hardness generally decreases bioavailability in aquatic invertebrates up to a hardness of 100 mg/l $CaCO_3$.

Redox conditions also affect bioavailability and in general metal bioavailability goes down with redox potential (Eh) or ORP. At circumneutral pH an Eh or ORP less than 0 volts results in reduced bioavailability especially in the presence of sulfur and sulfur compounds which precipitate metal sulfides. Some metals like iron and manganese are liberated at low Eh but these metals are not acutely toxic at low levels. Arsenic however can be liberated by the dissolution of iron and manganese oxides which tend to sorb the oxyanion form or arsenic and antimony.

11.3.1.4 Cation Exchange Capacity

CEC is the total capacity of a soil to hold exchangeable cations (i.e., essential nutrients) and provides a buffer against soil acidification. Soils with a higher clay fraction and organic matter tend to have a higher CEC than sandy soils. Toxicity to plants and earthworms from metals (particularly lead, cobalt, nickel, copper, and zinc) has been shown to increase with decreasing CEC of the soil (Organisation for Economic Co-operation and Development 2016, Oorts et al. 2021).

Geology can limit or enhance the bioavailability of metals as the specific mineral form of metals can control metal bioavailability. Metals that form strong bonds with silicate or oxide structures, for example, would be less bioavailable than soluble salt mineral forms. Weathering processes can release or sequester metals as ether solid or soluble forms. In general, highly weathered soils have lower bioavailability through water uptake because of binding to clays unless directly ingested in hand to mouth transfer. Hence solubility is a major factor in controlling bioavailability whereas solid state bioavailability is controlled by specific mineral or compound forms of metals. As soils or soil-like material is generally the most likely geologic solid media that could be ingested and given the exchangeability of metals in clay minerals the clay content of the soil is a good parameter for assessment of bioavailability. Soils with clays containing high cation exchange capacity of 50 milliequivalents per 100 g or more are more bioavailable than rocky or poorly

weathered soils. Organic rich soils often have CEC of 100 milliequivalents per 100 g and organic matter can increase bioavailability in general depending on soil pH.

The following table, prepared by the U.S.Navy (2000) presents a general overview of how different physical parameters in soil impact bioavailability potential of metals.

	Bioavailability		
Site History	**Low**	**Medium**	**High**
Metal Forms:			
Sulfides	X		
Elemental (metallic)	X		
Sulfates		X	
Carbonates			X
Oxides			X (except Ni)
Particle Size (of metal-bearing grains):			
Small			X
Large	X		
Weathering/Aging Time:			
Sulfides	X →		
Elemental	X →	→	
Carbonates		←	X
Oxides		←	X
Soil Chemistry			
pH:			
Acidic		X	
Basic			X (Cd, Hg, Pb, Ni)
Alkaline soils			X (Cd, Hg, Pb, Ni)
High TOC			X (Hg, Pb)
High Fe and Mn		X (As)	
Sulfide-producing soil		X (Cd, Hg, Pb, Ni)	

11.3.2 Discrete Surface Water Data

Surface water TAL metals (including mercury) data will be used to determine whether contaminants associated with AOC #16 – Area D (former Dumping Area) are being transported to the shoreline at concentrations that pose risk to human health and/or the environment. Background analytical data from Con Hook will be used to determine whether TAL metals (including mercury) detected in shoreline surface water at Iona Island are a result of DoD activities, or whether concentrations are consistent with/less than background (i.e., a result of naturally occurring conditions and/or contamination associated with the Hudson River and/or normal operations of the railroad).

Surface water hardness data will be used to calculate site-specific surface water screening values for metals. A total of 60 onsite and 40 background samples will be collected. The lower confidence limit (LCL) of the mean hardness values will be used as the hardness adjuster for water quality criteria. The use of the LCL will address variation and uncertainty in hardness data. One

value will be used for Iona Island (LCL of the 60 onsite hardness samples) and one value will be used for Con Hook (LCL of the 40 background hardness samples).

Surface water physical parameter data (pH, temperature, specific conductance, DO, ORP, turbidity, TSS, and salinity) will be used to help qualitatively support the risk assessments. Since total metals data in surface water will not be collected, TSS will provide an estimate of the amount of solid particulates in water to which metals could be bound. The amount of particulates is anticipated to be minimal due to the constant flushing of the river.

Background surface water hardness and physical parameter data (pH, temperature, specific conductance, DO, ORP, turbidity, TSS, and salinity) will be compared to onsite data to evaluate background representativeness and support the ERA.

11.3.3 Shoreline Sediment Metals Data

ISM sediment metals analytical results will be used to determine whether contaminants associated with AOC #16 – Area D (former Dumping Area) are present in sediment at concentrations that pose risk to human health and/or the environment. Background analytical data from Con Hook will be used to determine whether metals detected in shoreline sediment at Iona Island are a result of DoD activities, or whether concentrations are consistent with/less than background (i.e., a result of naturally occurring conditions and/or contamination associated with the Hudson River and/or normal operations of the railroad).

11.3.4 Shoreline Sediment Physical Parameter Data

Sediment physical parameter (grain size, TOC, ORP, and pH) data will be used to qualitatively evaluate bioavailability of metals in sediment and support the ERA (see Section 11.3.1 for discussions of each parameter). Physical parameter data will be used as screening tools to make estimates of solubility and contaminant availability. ISM sediment physical parameter data will also be used to evaluate background representativeness.

11.3.5 Shoreline Sediment AVS/SEM Data

The AVS/SEM approach developed by EPA (2005) will be used to conduct a screening level evaluation of toxicity to sediment-dwelling organisms. AVS/SEM is an approximation parameter used to account for differences in sulfide and refine predictions of heavy metal toxicity in sediments for the following divalent metals: Cu, Cd, Ni, Pb, and Zn. The equation is $(\Sigma SEM\text{-}AVS)/f_{oc}$. Results will be evaluated as follows:

- If $(\Sigma SEM\text{-}AVS)/f_{oc} < 130$ umol/g_{oc}, metals are unlikely to be bioavailable/toxic and there is no ecological risk.

- If 130 umol/g_{oc} < $(\Sigma SEM\text{-}AVS)/f_{oc} < 3000$ umol/g_{oc}, metals may be bioavailable/toxic and additional evaluation (i.e., toxicity testing) is recommended; ecological risk is unknown.

- If $(\Sigma SEM\text{-}AVS)/f_{oc} > 3000$ umol/g_{oc}, metals are likely bioavailable/toxic; however additional evaluations (i.e., toxicity testing) should be conducted to confirm that ecological risks exist.

If the screening level of toxicity indicates that metals may be bioavailable/toxic, and if additional sediment physical parameters also indicate that metals in sediment are bioavailable, then additional data will be collected to further evaluate risk to ecological receptors as part of the BERA.

11.4 WHAT TYPE OF DATA ARE NEEDED (MATRIX, TARGET ANALYTES, ANALYTICAL GROUPS, FIELD SCREENING, ONSITE ANALYTICAL OR OFFSITE LABORATORY TECHNIQUES, SAMPLING TECHNIQUES)?

Environmental media to be sampled include surface water and sediment at Iona Island, background surface water and sediment at Con Hook, and onsite soil at Iona Island. The Phase II investigation areas and sampling locations are presented in the following figures:

- Figure 11-1—Upland and hydric terrestrial ISM surface soil sampling locations (DU-4, DU-7, DU-9, DU-11, DU-13, DU-17, DU-18, and DU-19)

- Figures 11-2a and 11-2b—Iona Island surface water, ISM sediment, and discrete sediment AVS/SEM sampling locations

- Figure 11-3— Con Hook background surface water and ISM sediment sampling locations.

Data needs for the Phase II investigation are presented in Worksheet #17, which includes sample matrices, target analytes/analytical groups, onsite field screening and offsite laboratory analytical methods, and sampling techniques.

11.5 HOW GOOD DO THE DATA NEED TO BE TO SUPPORT THE ENVIRONMENTAL DECISION?

ISM surface soil data must be good enough to evaluate bioavailability of metals to terrestrial ecological receptors, support risk assessments, and support decision making.

The discrete surface water and ISM sediment metals analytical data must be good enough to support two important decisions: (1) are site concentrations greater than risk-based screening levels, and (2) are site concentrations greater than background concentrations? In addition, onsite and background surface water physical parameter data must be good enough to evaluate whether background locations are representative of onsite conditions.

ISM sediment physical parameter data must be good enough to qualitatively evaluate bioavailability to aquatic ecological receptors, support risk assessments, and support decision

making. In addition, onsite and background sediment physical parameter data must be good enough to evaluate whether background locations are representative of onsite conditions.

Discrete soil AVS/SEM sediment data must be good enough to assess the potential for toxicity to sediment-dwelling organisms within each onsite Phase II sediment DU, support the ERA, and support decision making.

11.5.1 Sources of Error

Total study error potential is equally attributable to sampling and measurement error because of the steps and sample volume associated with the planned sample collection and analysis. Successfully managing the magnitude of total study error is the result of understanding the error sources, generating an appropriate sampling design, and choosing accurate measurement techniques. The approaches used to manage study error for the planned sampling and analysis is discussed below.

- ***ISM Surface Soil Data***—The source of decision error for these results are equally attributable to sampling or measurement error. This conclusion is based upon review of the sampling and analysis strategy. The sampling design includes eight approximately 1-acre (e.g., 200 x 200 ft) DUs established during Phase I (5 upland soil and 3 hydric soil DUs), each sampled in triplicate, but the ISM sampling process is straightforward. ISM processing will be performed by a laboratory acceptable to the USACE Project Chemist. TOC and grain size analyses will be performed using the services of a DoD ELAP-accredited laboratory with standard methods. CEC analysis will be performed using a laboratory that is acceptable to the USACE Project Chemist, and QA/QC requirements per the DoD QSM, Version 5.3 (DoD 2019) will be followed to the extent possible

- ***Surface Water Sampling Data***—The source of decision error for these results are equally attributable to sampling or measurement error. This conclusion is based upon review of the sampling and analysis strategy. The sampling design includes multiple sampling locations, but the process is straightforward. The TAL metals and mercury analyses will be performed using the services of a DoD ELAP-accredited laboratory with standard methods. Hardness will be calculated using the services of a NELAP-accredited laboratory that is also acceptable to the USACE Project Chemist.

- ***ISM Sediment Data***—The source of decision error for these results are equally attributable to sampling or measurement error. This conclusion is based upon review of the sampling and analysis strategy. The sampling design includes three ½-acre sized sediment DUs at Iona Island, each sampled in triplicate, and 16 total 0.15-acre sized background SUs at Con Hook (8 on the Hudson River shoreline and 8 in Con Hook Marsh). Individual ISM samples will be collected from each background SU. The ISM sampling process is straightforward. ISM processing will be performed by a laboratory acceptable to the USACE Project Chemist. The metals, TOC, and grain size analyses will be performed using the services of DoD ELAP-accredited laboratories with standard methods.

- ***Discrete Sediment Data***—The source of decision error for AVS/SEM results are equally attributable to sampling or measurement error. This conclusion is based upon review of the sampling and analysis strategy. The sampling design includes 12 sampling locations (6 along the Iona Island Hudson River shoreline and 6 along the Iona Marsh shoreline), but the sampling process is straightforward. AVS/SEM analysis will be performed using the services of a DoD ELAP-accredited laboratory with standard methods.

11.5.2 Background Decision Error and Potential Consequences

The acceptable limits for false positive or false negative decision errors for metals concentrations in sediment as compared to background concentrations will be based on evaluating the potential consequences of decision errors (risks to human health and/or the environment) if specific contaminants are detected or are not detected above background concentrations obtained from Con Hook. Two potential decision errors could be made based upon interpreting sampling and analytical data:

1. Concluding that the concentration of a specific metals analyte that exceeds EPA screening values is below the background concentration obtained from Con Hook when it truly is above the background concentration.

2. Concluding that the concentration of a specific metals analyte that exceeds EPA screening values is greater than the background concentration obtained from Con Hook when it truly is below the background concentration.

The consequences of the first error would result in contamination being left in place, potentially posing risk to human and ecological receptors. The consequences of the second error would result in unnecessary expenditure, and diversion of resources that could be used for cleanup of other contaminated areas.

A baseline condition is established such that the concentration exceeding EPA screening values is less than the background concentration. The baseline condition is defined as the null hypothesis (H_0). The alternative is defined as the alternative hypothesis (H_a). This may be summarized as follows:

H_0: [concentration] = background concentration.
H_a: [concentration] > background concentration

A false positive error, also known as a Type I error, occurs when H_0 is falsely rejected (i.e., the sample data shows that the concentration of a metals analyte exceeding its EPA screening values also exceeds above the background concentration when it actually is below the background concentration). The measurement of the size of this error is called alpha (α), the level of significance. Alpha is expressed numerically as a probability or the tolerance for uncertainty.

A false negative error, also known as a Type II error, occurs when the H_0 is falsely accepted (i.e., the sample data shows that that the concentration of a metals analyte exceeding its EPA screening values is below the background concentration when it actually exceeds the background concentration. The measurement of the size of this error is called beta (β), or the complement of the power of the hypothesis test.

The tolerance limits for decision error have been established at α=5 % or 0.05 for false positives and β=20% or 0.2 for false negatives.

11.6 HOW MUCH DATA ARE NEEDED (NO. OF SAMPLES FOR EACH ANALYTICAL GROUP, MATRIX, AND CONCENTRATION)?

The number of samples for each analytical group and matrix planned to be collected during Phase II is presented in Worksheets #17 and #18 of this UFP-QAPP Addendum. Field QC samples intended to provide an indication of the consistency of sample collection and analyses are listed on Worksheet #20 of this UFP-QAPP Addendum.

Field QC samples will include duplicate samples and matrix spike (MS)/matrix spike duplicates (MSD) collected at a rate of 1 per 20 samples as required by the analytical method. While neither field duplicates nor MS/MSDs are needed to evaluate precision for ISM or discrete sample designs that support statistical methods for making inferences about contamination, the laboratory requires MS/MSDs for both the discrete and ISM samples for the analytical method. Duplicates will be collected with the discrete surface water samples to support assessment of variability in surface water concentrations, although statistical evaluation will also be performed. The analytical laboratory will also perform MS/MSD on discrete surface water and ISM soil and sediment samples at a rate of 1 per 20 samples as required by the analytical method. MS/MSD for ISM samples will require additional volume (approximately 1.5 kg rather than 1 kg). The precision from the ISM process will be used when assessing the data.

11.7 WHO WILL COLLECT AND GENERATE THE DATA?

EA field personnel will conduct the environmental sampling. Laboratory analysis will be conducted as follows:

- ISM sediment and soil sample processing will be completed by Eurofins TestAmerica Canton, OH.
- Laboratory analytical services for surface water, sediment, and soil samples will be provided by the following:
    - Eurofins TestAmerica Denver, CO: surface water and ISM sediment sample metals and mercury analyses and surface water hardness calculation

- Eurofins Lancaster Laboratories, Inc., PA: discrete sediment sample AVS/SEM analysis

- Katahdin Analytical Services, LLC, located in Scarborough, ME: ISM soil and sediment sample TOC and grain size analyses

- Eurofins TestAmerica, Corpus Christi, TX: ISM soil sample CEC analysis

The analytical laboratories will generate portable document format (PDF) laboratory reports and upload Stage 2A staged electronic data deliverables (SEDDs) to FUDSChem. Validation of data generated by the analytical laboratories will be performed by the EA Project Chemist and certified by the USACE Project Chemist. Data validation procedures are presented in Worksheet #36 of this UFP-QAPP Addendum. Data usability of validated analytical data and field screening data will be evaluated by the EA Project Chemist and EA Data Manager. Non-chemistry data sets will not undergo the same type of usability analysis as analytical chemistry data (e.g., precision, accuracy, representativeness, completeness, comparability, sensitivity analysis), but will be evaluated for usability, the results of which will be included in the RI report (e.g., geospatial, physical testing, hydraulic, field water quality, and field screening data).

11.8 WHERE, WHEN, AND HOW SHOULD THE DATA BE COLLECTED/ GENERATED?

Where: The Phase II DUs and sampling locations are presented on Figures 11-1 and 11-2a/b. Con Hook background surface water sampling locations and sediment SUs are presented in Figure 11-3. Additional details, including coordinate information, are provided in Worksheet #18.

When: The Phase II field work is scheduled to commence in late winter / early spring 2022. The project schedule is presented in Worksheet #14 and #16.

While the investigation areas are generally accessible, features and conditions may result in constraints on individual sampling locations. Time constraints for sampling include weather conditions and tidal fluctuations. Sampling will not be conducted during storm events due to health and safety concerns.

There are no additional time constraints for ISM soil sampling in the upland portion of Iona Island (surface soil at DU-4, DU-7, DU-9, DU-11, and DU-13). DU-17, DU-18, and DU-19 are inundated during high tide. Therefore, ISM hydric soil sampling at will be conducted during low tide.

Surface water and sediment samples will not be collected during precipitation events as input from precipitation and surface water runoff can dilute samples. Additional time constraints for surface water and sediment sampling are as follows:

- Surface water sampling within AOC #16 – Area D (former Dumping Area) will be conducted during tidal inundation (rising or high tide) to capture potential metals leaching from soil to surface water.

- Surface water sampling at the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area) will be conducted during high or receding tide to capture water flowing from AOC #16 – Area D (former Dumping Area).

- Surface water sampling at outlets of the riprap filled drainage swale and tidal wetland swale will be conducted during receding tide to capture potential flow from AOC #16 – Area D (former Dumping Area).

- A boat will be required to access Con Hook. Surface water sampling at Con Hook (CHHRS-SW-01 through CHHRS-SW-20 and CHMS-SW-01 through CHMS-SW-20) will be collected at low tide concurrent with sediment sampling to limit the number of trips to and time spent at Con Hook.

- Sediment sampling along the Iona Island and Con Hook shorelines will be conducted during low tide when sediment is exposed.

How: Further detail on the field tasks and methods is presented in Worksheet #14 and #16. Field SOPs are listed in Worksheet #21. Analytical sampling SOPs are listed in Worksheet #23.

11.9 HOW WILL THE DATA BE REPORTED?

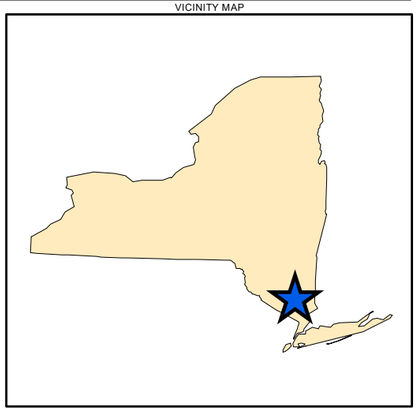
Once a SEDD is successfully uploaded and certified by the analytical laboratory in FUDSChem, an Automated Data Review (ADR) process will be automatically initiated. The ADR will perform a Stage 2A data validation by comparing the SEDD to the approved eQAPP, summarizing QC outliers in an ADR report, and applying data validation qualifiers to associated results. The "first review," EA chemist will perform a Stage 2B data validation based on the initial ADR Stage 2A validation. The data validator will review the ADR report against the PDF laboratory report to (1) verify/modify the ADR qualification of the sample results and to (2) supplement the ADR review with a manual review of QC elements that were not included in the ADR review. The data validator will complete the data review checklist and prepare a data validation report that summarizes the data validation findings. The "second reviewer," EA chemist will review the data validation report and sign-off as the second reviewer. The data validation report will then be uploaded to the FUDSChem library.

11.10 HOW WILL THE DATA BE ARCHIVED?

Data will be managed in the FUDSChem per the requirements specified in the USACE New England District (NAE) Data Management Plan (USACE NAE 2016).

Data will be transferred to the project stakeholders upon completion of the project. Retrieval of data by others will be at the discretion of the project stakeholders. The length of time that records will be archived will be at the discretion of the project stakeholders.

VICINITY MAP



Legend

- FUDS Boundary
- Phase II Soil Physical Parameter DU
- Upland
- Marsh/Wetland

0 200 400
Feet
1 in = 200 ft

N

FIGURE 11-1
Phase II Surface Soil Physical Parameter Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial: EA Engineering, Science, and Technology. April, 7 2020
AOC Boundary: USAGC 2018
Upland and Marsh/Wetland: 2020 EA Shoreline Reconnaissance

Map Date: 1/31/2022

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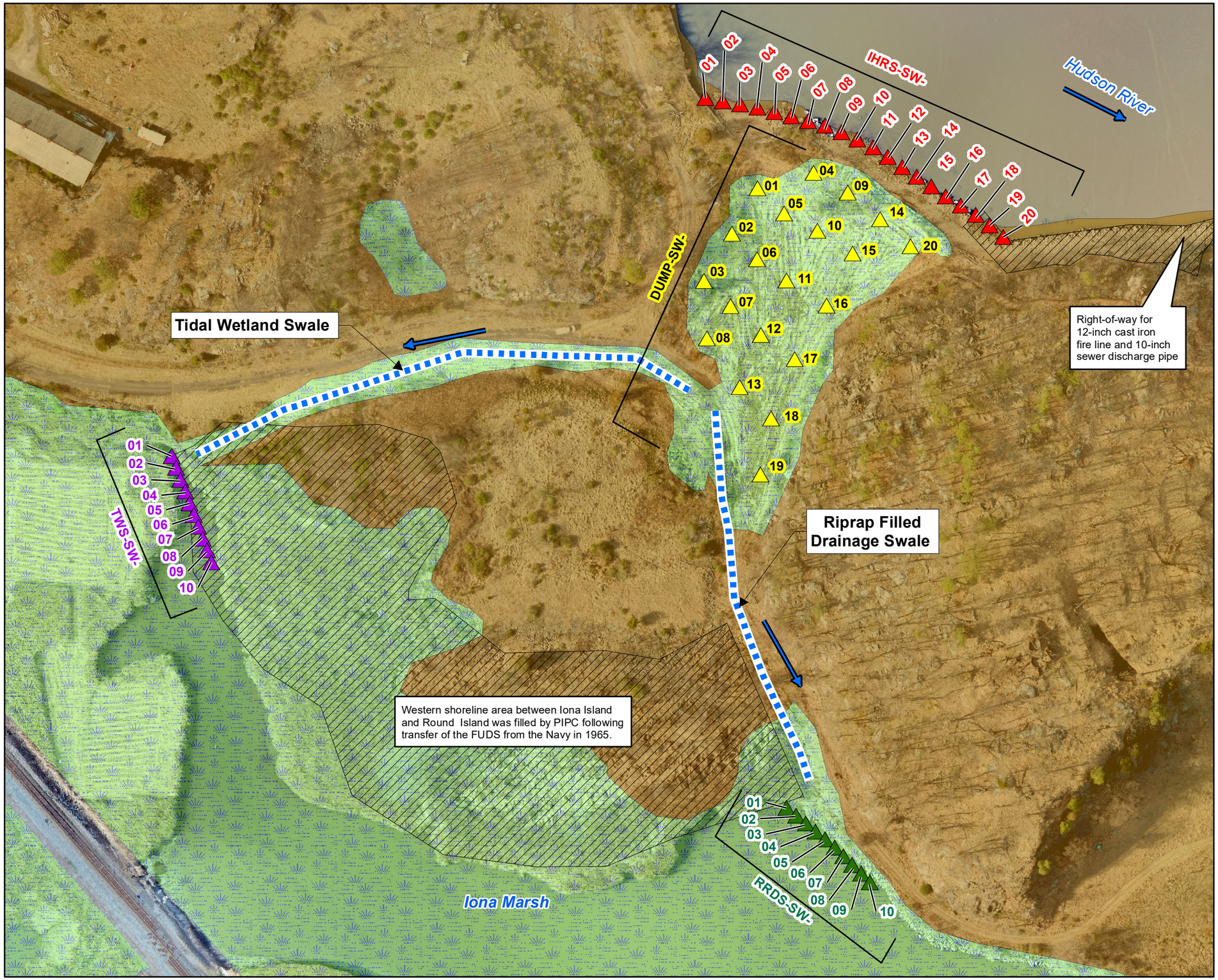

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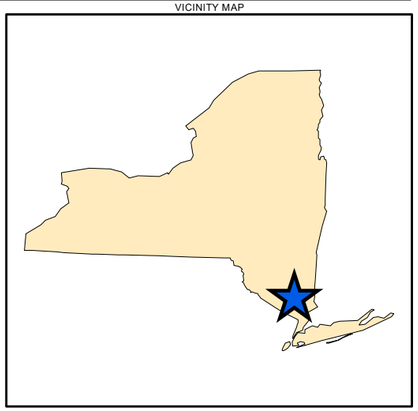

Legend

- AOC #16 – Area D (former Dumping Area)
- Upland
- Marsh/Wetland
- ISM Sediment Decision Unit
- Surface Water Migration Pathway

Surface Water Sampling Location

- Iona Island Hudson River Shoreline
- AOC #16 – Area D (former Dumping Area)
- Iona Marsh Tidal Wetland Swale Outlet
- Iona Marsh Riprap Filled Drainage Swale Outlet
- Surface Water Flow Direction

0 100 200
Feet
1 in = 100 ft

N

FIGURE 11-2a
Phase II Iona Island Surface Water Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial and Topographic Contours: EA Engineering, Science, and Technology. April, 7 2020
Disturbed Ground/Fill Area and AOC Boundaries: USAGC 2018
Upland and Marsh/Wetland: 2020 EA Shoreline Reconnaissance
Map Date: 2/7/2022

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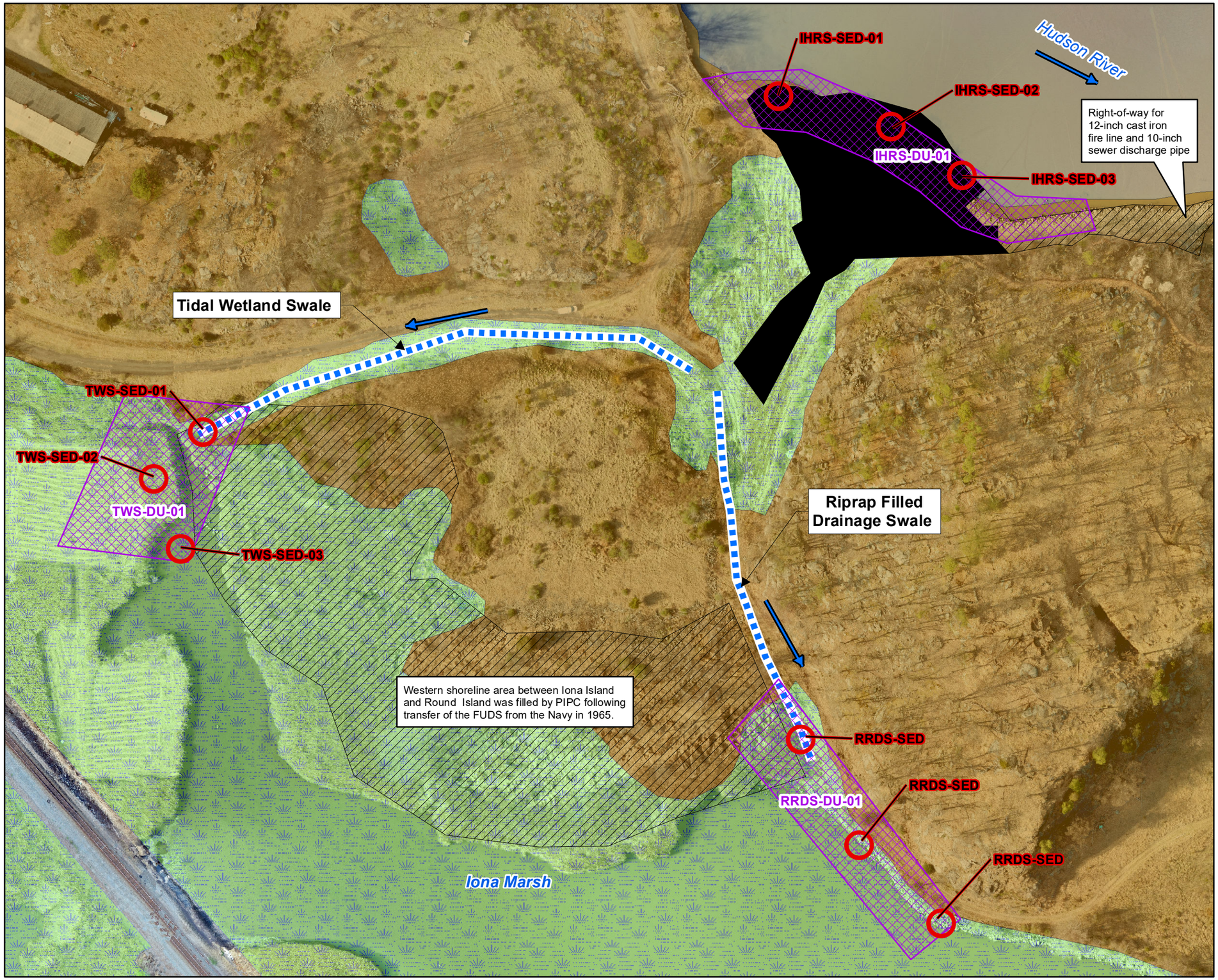

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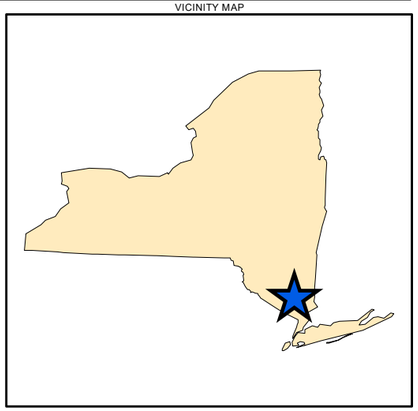

Legend

- AOC #16 – Area D (former Dumping Area)
- Upland
- Marsh/Wetland
- ISM Sediment Decision Unit
- Surface Water Migration Pathway
- Sediment AVS/SEM Sampling Location
- Surface Water Flow Direction

0 100 200
Feet
1 in = 100 ft

N

FIGURE 11-2b
Phase II Iona Island Sediment Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial and Topographic Contours: EA Engineering, Science, and Technology. April, 7 2020
Disturbed Ground/Fill Area and AOC Boundaries: USAGC 2018
Upland and Marsh/Wetland: 2020 EA Shoreline Reconnaissance
Map Date: 2/7/2022

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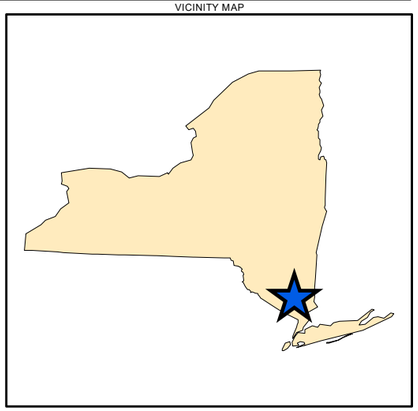
VICINITY MAP



Legend

- Background Sediment Sampling Unit
- Discrete Surface Water Sampling Location
- Surface Water Flow Direction

0 200 400
Feet
1 in = 200 ft

N

FIGURE 11-3
Con Hook Background Surface Water and Sediment Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Source
Aerial: ESRI ArcGIS Online Map Service (ESRI 2009b).
Service Layer Credits: Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community

Map Date: 2/7/2022

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Table 11-1 Sources for Ecological Screening Values for Freshwater

			Ecological Screening Values for Freshwater[a]			
Analyte	**CAS No.**	**NRWQC (current values) (µg/L)**	**EPA BTAG Region 3 Freshwater Screening Values 2006 (µg/L)**	**Source**	**EPA Region 4 Ecological Screening Levels Freshwater Chronic 2018 (µg/L)**	**Source**
Aluminum	7429-90-5	NA	87	NRWQC 2004	**87**	NRWQC
Antimony	7440-36-0	NA	30	Suter et al. 1996	**190**	GLI
Arsenic	7440-38-2	**150**	5	CCME	150	NRWQC
Barium	7440-39-3	NA	4	Suter et al. 1996	**220**	GLI
Beryllium	7440-41-7	NA	0.66	Suter et al. 1996	**11**	GLI
Cadmium	7440-43-9	**0.72**	0.25	NRWQC 2004	0.45	NRWQC
Calcium	7440-70-2	NA	116,000	Suter et al. 1996	**116,000**	Suter et al. 1996
Chromium, Total	7440-47-3	**74**	85	NRWQC 2004	42	NRWQC
Cobalt	7440-48-4	NA	23	Suter et al. 1996	**19**	GLI
Copper	7440-50-8	NA	9	NRWQC 2004	**4.95**	NRWQC
Iron	7439-89-6	**1,000**	300	CCME	1,000	NRWQC
Lead	7439-92-1	**2.5**	2.5	NRWQC 2004	1.25	NRWQC
Magnesium	7439-95-4	NA	82,000	Suter et al. 1996	**82,000**	Suter et al. 1996
Manganese	7439-96-5	NA	120	Suter et al. 1996	**93**	GLI
Mercury	7439-97-6	**0.77**	0.026	CCME	0.77	NRWQC
Nickel	7440-02-0	**52**	52	NRWQC 2004	28.9	NRWQC
Potassium	7440-09-7	NA	53,000	Suter et al. 1996	**53,000**	Suter et al. 1996
Selenium	7782-49-2	NA	1	CCME	**5.0**	NRWQC
Silver	7440-22-4	**1.6**	3.2	NRWQC 2004	0.06	NRWQC
Sodium	7440-23-5	NA	680,000	Suter et al. 1996	**680,000**	Suter et al. 1996
Thallium	7440-28-0	NA	0.8	CCME	**6.0**	GLI
Vanadium	7440-62-2	NA	20	Suter et al. 1996	**27**	GLI
Zinc	7440-66-6	**120**	120	NRWQC 2004	66	NRWQC

Notes:
NA = not available
a. Ecological screening values for freshwater, chronic exposure. Hierarchy for selected values is as follows:
1) NRWQC, current values. available: https://www.epa.gov/wqc/national-recommended-water-quality-criteria-aquatic-life-criteria-table
2) Great Lakes Initiative (GLI), Clearinghouse Resources Tier II criteria revised 2013. Available: https://www.epa.gov/gliclearinghouse. These values were selected over Canadian Council of Ministries of the Environment (2003), per USACE NAE.
3) Lastly, values from the publication by Suter et al. 1996. Toxicological benchmarks for screening potential contaminants of concern for effects on aquatic biota: 1996 Revision. ES/ER/TM-96/R2. https://rais.ornl.gov/documents/tm96r2.pdf. This source was used when no other values were available.

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UFP-QAPP ADDENDUM WORKSHEET #12 – MEASUREMENT PERFORMANCE CRITERIA TABLES

This worksheet documents the quantitative measurement performance criteria in terms of precision, bias, and sensitivity for laboratory measurements and is used to guide the selection of appropriate measurement techniques and analytical methods. Measurement performance criteria are developed to ensure collected data will satisfy the DQOs documented on Worksheet #11. Measurement performance criteria from Phase I that are applicable to Phase II are provided in the UFP-QAPP (EA 2020a). Measurement performance criteria for additional Phase II parameters are provided in this UFP-QAPP Addendum. The following table presents the location of measurement performance criteria tables for each Phase II media and laboratory analysis.

Note:

- Eurofins TestAmerica Denver measurement performance criteria tables for TAL metals in soil/sediment by EPA Methods 6010C/6020A and mercury in soil/sediment by 7471B are presented in the UFP-QAPP (EA 2020a)

- Hardness by 2340B is a calculation method and there are no applicable measurement performance criteria tables for hardness

- The 6010C metals tables for water presented in this UFP-QAPP Addendum are applicable to the hardness calculation results

- Katahdin Analytical Services, LLC measurement performance criteria tables for TOC by Walkley-Black are presented in the UFP-QAPP (EA 2020a)

- There are no measurement performance criteria tables for grain size by ASTM D422.

- Measurement performance criteria for global positioning system (GPS) surveys are included in this UFP-QAPP Addendum.

Analytical Method	Location of Measurement Performance Criteria Table
Surface Water Analyses	
TAL Metals – Method 6010C	Table 12-1 of this UFP-QAPP Addendum
TAL Metals – Method 6020A	Table 12-2 of this UFP-QAPP Addendum
Mercury – Method 7470A	Table 12-3 of this UFP-QAPP Addendum
Hardness – SM2340B	NA
Sediment Analyses	
Metals – Method 6010C	Table 12-1 of the UFP-QAPP (EA 2020a)
Metals – Method 6020A	Table 12-2 of the UFP-QAPP (EA 2020a)
Mercury – Method SW7471	Table 12-3 of the UFP-QAPP (EA 2020a)
Sulfide (for AVS/SEM) by SM 4500-S2D-2011	Table 12-4 of this UFP-QAPP Addendum
Metals – ICP Water (for AVS/SEM) by SW-846 6010C	Table 12-5 of this UFP-QAPP Addendum
TOC – Method Walkley-Black	Table 12-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	NA
Soil Analyses	
TOC –Method Walkley-Black	Table 12-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	NA
CEC by SW-846 Method 9081	Table 12-6 of this UFP-QAPP Addendum
Global Positioning System Surveys	
Global Positioning System Surveys	Table 12-7 of this UFP-QAPP Addendum

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
CEC = Cation-exchange capacity
ICP = Inductively coupled plasma
NA = Not applicable
SEM = Simultaneously extracted metals
SM = Standard method
QAPP = Quality Assurance Project Plan
TAL = Target Analyte List
TOC = Total organic carbon
UFP = Uniform Federal Policy

Table 12-1 Measurement Performance Criteria TAL Metals – Water Method 6010C (Eurofins TestAmerica Denver)

Matrix	Water
Analytical Group	TAL Metals (Eurofins TestAmerica Denver)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance[3]	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	SW6010/ DV-MT-0021	Completeness	100%	NA	S&A
		Precision	RPD <50% for discrete samples	Field duplicate discrete samples	S&A
		Accuracy/bias/ contamination	No target analyte ≥ ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater	Blanks (method and instrument)	S&A
		Accuracy/bias	90 to 110%R	ICV and CCV	A
		Accuracy/bias	80 to 120%R	Low-level calibration check (only if one-point calibration is used)	A
		Accuracy/bias	ICS-A: the non-spiked analytes < LOD, ICS-AB: within ±20% of expected value	ICS	A
		Accuracy	%R per DoD QSM 5.3, Table C-3/C-4	LCS, MS, and MSD	A
		Precision	RPD<20%	MS and MSD	A
		Accuracy/bias	5-fold dilution within ±10% of original value for results > 50x LOQ	Serial dilution	A
		Accuracy/bias	Within ±20% of expected value for results < 50x LOQ	Post-digestion spike	A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.
3) MS/MSDs will be performed on soil samples at a rate of 1 per 20 samples as required by the analytical method.

NOTES:
% = Percent
%R = Percent recovery
CCV = Continuing calibration verification
DoD = Department of Defense
DU = Decision Unit
ICP = Inductively coupled plasma
ICS = Interfering element check standards
ICS-A = Interference check solution A
ICS-AB = Interference check solution AB
ICV = Initial calibration verification
LCS = Laboratory control sample
LOD = Limit of detection
LOQ = Limit of quantitation
MS = Matrix spike
MSD = Matrix spike duplicate
NA = Not applicable
QC = Quality control
QSM = Quality Systems Manual
RPD = Relative percent difference
SOP = Standard operating procedure

Table 12-2 Measurement Performance Criteria TAL Metals – Water Method 6020A (Eurofins TestAmerica Denver)

Matrix	Water
Analytical Group	TAL Metals (Eurofins TestAmerica Denver)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance[3]	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-007 EA-SOP-043	SW6020/DV-MT-0018	Completeness	100%	NA	S&A
		Precision	RPD <50% for discrete samples	Field duplicate discrete samples	S&A
		Accuracy/bias/ contamination	No target analyte ≥ ½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater	Blanks (method and instrument)	S&A
		Accuracy/bias	90 to 110%R	ICV and CCV	A
		Accuracy/bias	80 to 120%R	Low-level calibration check (only if one-point calibration is used)	A
		Accuracy/bias	ICS-A: the non-spiked analytes < LOD, ICS-AB: within ±20% of expected value	ICS	A
		Accuracy	%R per DoD QSM 5.3, Table C-5/C-6	LCS, MS, and MSD	A
		Precision	RPD<20%	MS and MSD	A
		Accuracy/bias	5-fold dilution within ±10% of original value for results > 50x LOQ	Serial dilution	A
		Accuracy/bias	Within ±20% of expected value for results < 50x LOQ	Post-digestion spike	A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.
3) MS/MSDs will be performed on soil samples at a rate of 1 per 20 samples as required by the analytical method.

NOTES:
% = Percent
%R = Percent recovery
CCV = Continuing calibration verification
DoD = Department of Defense
ICS-A = Interference check solution A
ICS-AB = Interference check solution AB
ICV = Initial calibration verification
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
SOP = Standard operating procedure

Table 12-3 Measurement Performance Criteria Mercury – Water Method 7470A (Eurofins TestAmerica Denver)

Matrix	Water
Analytical Group	Mercury (Eurofins TestAmerica Denver)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance3	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-007 EA-SOP-043	SW7470A/ DV-MT-0017	Completeness	100%	NA	S&A
		Precision	RPD ≤50% for discrete samples	Field duplicate discrete samples	S&A
		Accuracy/bias/ contamination	No target analyte ≥ ½ LOQ or ≥1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater	Blanks (method and instrument)	S&A
		Accuracy/bias	90 to 110%R	ICV and CCV	A
		Accuracy/bias	80 to 120%R	Low-level calibration check (if one-point calibration)	A
		Accuracy	%R per DoD QSM 5.3, Table C-11/C-12	LCS, MS, and MSD	A
		Precision	RPD≤20%	MS and MSD	A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.
3) MS/MSDs will be performed on soil samples at a rate of 1 per 20 samples as required by the analytical method.

NOTES:
% = Percent.
%R = Percent recovery
CCV = Continuing calibration verification
DoD = Department of Defense
ICV = Initial calibration verification
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
SOP = Standard Operating Procedure

Table 12-4 Measurement Performance Criteria – Sulfide (for AVS/SEM) by SM 4500-S2D-2011 (Eurofins Lancaster Laboratories, Inc.)

Matrix	Sediment AVS/SEM extract
Analytical Group	Sulfide (Eurofins Lancaster Laboratories, Inc.)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-016 EA-SOP-021	SM 4500-S2D-2011 / WI11483	Accuracy/Bias/Precision	57-108% %R, RPD≤20%	Laboratory Control Spike/Matrix Spike and their Duplicates	A
		Accuracy/Laboratory Contamination	No analytes detected > 1/2 LOQ or >1/10 the amount measured in any sample	Method Blank	A
		Precision	RPD≤20%	Lab Duplicate	S & A
		Precision	RPD<50%	Field Duplicate	S & A
		Completeness	100%	Reported Sample Data	S & A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.

NOTES:
LCS = Laboratory control sample
LOQ = Limit of quantitation
PAL = Project action limit
QC = Quality control
RPD = Relative percent difference
SM = Standard method
SOP = Standard Operating Procedure

Table 12-5 Measurement Performance Criteria – Metals – ICP Water (for AVS/SEM) by SW-846 6010C (Eurofins Lancaster Laboratories, Inc.)

Matrix	Sediment AVS/SEM extract
Analytical Group	Metals – ICP (Eurofins Lancaster Laboratories, Inc.)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-016 EA-SOP-021	SW-846 6010C / WI11931	Accuracy/Bias/Precision	%R per DoD QSM 5.3, Table C-3/C-4; RPD ≤20%	Laboratory Control Spike/Matrix Spike and their Duplicates	A
		Accuracy/Laboratory Contamination	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample	Method Blank	A
		Precision	RPD ≤20%	Lab Duplicate	S & A
		Precision	RPD <50%	Field Duplicate	S & A
		Completeness	100%	Reported Sample Data	S & A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.

NOTES:
% = Percent
DoD = Department of Defense
LCS = Laboratory control sample
LOQ = Limit of quantitation
PAL = Project action limit
QC = Quality control
QSM = Quality systems manual
RL = Reporting limit
RPD = Relative percent difference

Table 12-6 Measurement Performance Criteria – Cation Exchange Capacity by SW-846 Method 9081 (Eurofins TestAmerica Corpus Christi)

Matrix	Sediment
Analytical Group	CEC
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-016 EA-SOP-021	SW-846 Method 9081 and Method 6010B (SOP CC-ATM-M005, SOP CC-ATM-M020)	Accuracy, Precision	87-115% recovery; MSD: RPD ≤ 20%	Matrix Spike and Duplicate	A
		Accuracy	87-115% recovery	Laboratory Control Sample	A
		Accuracy/Laboratory Contamination	No detections >1/2 LOQ or >1/10 the amount measured in any sample	Method Blank	A
		Completeness	100%	Reported Sample Data	S&A

1) Referenced SOPs for sampling procedures are presented in Worksheet 21.
2) Referenced SOPs for the analytical method are presented in Worksheet 23.

NOTES:
% = Percent
MSD = Matrix spike duplicate
QC = Quality control
RL = Reporting limit
RPD = Relative percent difference

UFP-QAPP ADDENDUM WORKSHEET #14 and #16 – PROJECT TASKS AND SCHEDULE

This worksheet lists the project-specific sampling tasks to be performed in support of the Iona Island Ammunition Depot FUDS HTRW RI. A general project schedule presenting the tasks and planned start and end dates is included at the end of this worksheet. The schedule will be modified as necessary during the field activities based on weather or any access issues.

Phase II field tasks will be conducted in accordance with this UFP-QAPP Addendum. The sampling design and rationale is presented in detail in Worksheet #17 of this UFP-QAPP Addendum. Field SOPs used during the Phase I investigation that are applicable to Phase II are presented in Appendix H of the UFP-QAPP (EA 2020a). Additional field SOPs for Phase II are listed in Worksheet #21 and presented in Appendix D of this UFP-QAPP Addendum. Analytical SOPs that are applicable to Phase II are listed in Worksheet #23 and presented in Appendix E of this UFP-QAPP Addendum.

14.1 FUDSCHEM DATA MANAGEMENT – EA

Prior to field sampling, the Phase I eQAPP prepared within FUDSChem will be updated. The eQAPP will be fully compliant with the UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum, incorporating methods, sample types, and quality specifications (LOD, LOQ, Holding Time, QC Sample Acceptance Limits, etc.) for Phase II. The pre-population of sampling event information (location information, sample type, matrix, methods, etc.) will also be performed to ensure accurate database field matching and status tracking within FUDSChem.

14.2 MOBILIZATION AND DEMOBILIZATION TASKS – EA, SUBCONTRACTORS

Mobilization/demobilization will occur based on the proposed field schedule. Mobilization tasks will include the following:

- Notify USACE and PIPC prior to mobilizing equipment and field personnel to the site.
- Coordinate with field personnel and subcontractors as needed; ensure staff have proper certification (e.g., Occupational Safety and Health Administration, DoD Explosives Safety TP-18).
- Procure field equipment and supplies.
- Identify and set up office space and equipment staging/storage areas, as needed.
- Order sample bottles and field monitoring equipment. Sample bottle requirements are presented in Worksheet #19 and #30 of this UFP-QAPP Addendum. The equipment necessary to execute the field work and complete the project tasks is detailed below, and in the SOPs listed in Worksheet #21 of this UFP-QAPP Addendum.

- Mobilize field staff to the site.

Demobilization will include removing field equipment and supplies, returning rented equipment, performing general cleanup, and organizing and finalizing field documentation.

14.3 SITE SPECIFIC TRAINING – EA, SUBCONTRACTORS

A field kickoff meeting will be conducted during mobilization to provide site-specific training to the field personnel. The field kickoff meeting may include the following attendees: USACE, EA, and PIPC (to assist with natural resource and cultural resource awareness training if necessary).

The field team will review the following:

- Project plans (i.e., UFP-QAPP [EA 2020a], this UFP-QAPP Addendum, Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP) presented in Appendix A of the UFP-QAPP (EA 2020a)

- PIPC expectations and code of conduct for the field team during RI activities

- Site hazards/site safety and health concerns, evacuation plans and procedures/route to the hospital

- Safety procedures

- Procedures to minimize environmental impacts

- Procedures to identify natural or cultural resources

- Procedures to be employed if/when natural or cultural resources are identified; an Environmental Protection Plan is provided in Appendix B of the UFP-QAPP (EA 2020a).

14.4 HEALTH AND SAFETY TASKS – EA, SUBCONTRACTORS

Phase II field activities will be conducted in accordance with the APP/SSHP presented in Appendix A of the UFP-QAPP (EA 2020a). Each team member will read and sign the USACE-accepted APP/SSHP and receive site-specific training that covers site safety, emergency procedures, QC (pass/fail criteria), general daily activities protocols, site communication, and general site layout including safe areas, and natural resource/environmental and cultural/ archaeological concerns.

Entrance briefings and safety meetings will be conducted prior to the start of fieldwork to familiarize the team personnel with site health and safety requirements, the objectives and scope of field activities, and chain-of-command. Personnel mobilized to the site will meet requirements

for Occupational Safety and Health Administration hazardous waste operations training, and medical surveillance requirements as specified in the APP/SSHP. Site personnel will also be trained to perform the specific tasks to which they are assigned. At no time will site personnel be tasked with performing an operation or duty for which they do not have appropriate training. The field team will be familiar with sample locations and will identify related field support areas and requirements.

14.5 INVESTIGATIVE TASKS – EA

Sampling will be completed by or under the full-time supervision of an EA field geologist. Field equipment including water quality meters and ORP meters will be calibrated daily prior to sampling activities, and calibration checks will be conducted at the end of each sampling day and when anomalous readings are obtained, in accordance with manufacturer recommendations. Calibration standards used will bracket the expected values for the parameter to be measured. If the calibration procedure for the method does not allow for the number of standards needed to bracket expected concentrations, then additional standards will be incorporated into a calibration check performed at the start of the day immediately after calibration. Field screenings and samples analyzed prior to a failing calibration check will be resampled and/or re-analyzed accordingly.

Calibration will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]). Criteria will be presented for the beginning and end-of-day calibration checks. Water quality meter calibration information will be recorded on field forms provided in Appendix F of this UFP-QAPP Addendum.

Sampling locations and DU/SU vertex coordinates are presented in Worksheet #18 of this UFP-QAPP Addendum. Sampling locations and DU/SU vertices will be located in the field with a hand-held GPS unit (to within sub-meter accuracy) and marked with a highly visible marker (i.e., high visibility flagging and a stake), prior to sample collection.

14.5.1 ISM Surface Soil Sampling Tasks

ISM surface soil samples will be collected from 0-6 in. bgs in accordance with EA-SOP-057 (Appendix D of this UFP-QAPP Addendum) and ITRC ISM (ITRC 2020). Each approximately 1-acre (e.g., 200 x 200 ft) sized DU will be sampled in triplicate (initial ISM sample plus two additional samples) as presented in Worksheet #11 to calculate the 95UCL.

Onsite soil DUs were established during Phase I. DU vertex coordinates are presented in Worksheet #18. DU boundaries will be located in the field with a hand-held GPS unit (typically to within sub-meter) and marked with a highly visible marker (i.e., high-visibility flagging and a stake), prior to sample collection.

Each surface soil incremental sample be comprised of 50 increments of equal volume. Worksheet #11 presents the decision criteria used to determine the No. of increments comprising each incremental sample. Increments will be collected using a dedicated incremental sampling tool

with plungers collecting soil equivalent to approximately 33.3 g in volume, which will provide a total weight of 1 kg of sediment. The depth of each increment will be approximately 6 in. bgs, which is the length of the incremental sampling tool's sampling plunger attachment. Each of the triplicate incremental samples (initial sample plus two additional samples) will be comprised of surface soil increments taken from different, randomly selected locations within the DUs.

ISM surface soil samples will be collected for analysis of the following parameters:

- TOC by Walkley-Black
- Grain size by ASTM D422 (sieve and hydrometer)
- CEC by EPA SW-846 Method 9081
- ORP by field screening (field and standard hydrogen electrode corrected values).

Each incremental sample will be collected in a laboratory-approved bag or container large enough to accommodate the total 1-kg volume for analysis. Analytical samples will be submitted to Eurofins TestAmerica Canton for ISM processing. TOC and grain size samples will then be submitted to Katahdin Analytical Services, LLC, located in Scarborough, ME for analysis. CEC samples will be submitted to Eurofins TestAmerica, Corpus Christi, TX for analysis.

ORP for soil samples will be determined in the field using an ORP meter. Approximately 20 g of well composited soil will be mixed with 20 milliliters (mL) of deionized water and thoroughly mixed and homogenized. The suspension will then be allowed to rest to allow most of the suspended clay to settle out from the suspension prior to measuring ORP. Both field and standard hydrogen electrode corrected values will be reported. ORP will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

14.5.2 Surface Water Sampling

Surface water sampling along the Hudson River shoreline of AOC #16 – Area D (former Dumping Area) and at Con Hook is dependent on tidal fluctuations. The tidal range averages approximately 2.9 ft (Yozzo et al. 2005). In addition, water depth observed within AOC #16 – Area D (former Dumping Area) during tidal inundation ranged from 1 to 2 ft. Water at AOC #16 – Area D (former Dumping Area) (DU-17-SW, DU-18-SW, and DU-19-SW) will be collected during tidal inundation (rising or high tide) to capture potential metals leaching from soil. Hudson River shoreline samples will be collected at high or receding tide to capture transport of metals to the shoreline via tidal flow.

A boat will be required to access Con Hook. Therefore, to limit the No. of trips to and time spent at Con Hook, background surface water samples at Con Hook will be collected at low tide concurrent with sediment sampling.

Surface water in the riprap filled drainage swale and tidal wetland swale leading from the western boundary of AOC #16 – Area D (former Dumping Area) to Iona Marsh will only be sampled if

water is observed within the drainages. If water is not observed in the drainages, then water samples will not be collected.

Surface water samples will be collected using discrete sampling methods presented in EA-SOP-007 (Appendix D of this UFP-QAPP Addendum). If the water is sufficiently deep, water will be collected by submerging a laboratory-provided bottle into the water without disturbing bottom sediment, and without overfilling sample bottles. Otherwise, surface water samples will be collected using a dedicated (disposable) dipper cup, or non-dedicated sampling equipment (dipper with stainless-steel sampler, or pond sampler) and transferred directly into pre-preserved bottles provided by the analytical laboratory. Non-dedicated sampling equipment will be decontaminated prior to initial usage and between sampling locations as presented under Equipment Decontamination Tasks.

Coordinates for sampling locations will be collected in the field with a hand-held GPS unit. Coordinate information will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]). Discrete surface water samples will be collected for analysis of the following parameters:

- Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A
- Dissolved (field filtered) mercury by EPA Method 7470A
- Hardness by SM2340.

Dissolved TAL metals (including mercury) samples will be collected by field filtering water through dedicated (disposable) 0.45-micron filters using a peristaltic pump and dedicated (disposable) sample tubing. Surface water samples for hardness and dissolved TAL metals (including mercury) will be submitted to Eurofins TestAmerica Denver, CO for offsite laboratory analysis.

Water quality parameters will be recorded at each surface water sampling location following sample collection using a submersible water quality meter capable of measuring each parameter (pH, temperature, specific conductance, DO, ORP, turbidity, TSS, and salinity). Data for each sampling location will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

14.5.3 Incremental Sampling Methodology Sediment Sampling Tasks

Incremental sampling will be conducted in accordance with EA-SOP-057 (Appendix D of this UFP-QAPP Addendum) and ITRC ISM (ITRC 2020). Each ½-acre sized Iona Island sediment DU will be sampled in triplicate (initial ISM sample plus two additional samples) as presented in Worksheet #11 to calculate the 95UCL. Single ISM samples will be collected from each 0.15-acre background sediment SU. ISM samples will be collected for analysis of the following parameters:

- Metals by EPA Methods 6010C/6020A

- Mercury by EPA Method 71471B.
- TOC by Walkley-Black
- Grain size by ASTM D422 (sieve and hydrometer)
- pH by field screening
- ORP by field screening (field and standard hydrogen electrode corrected values).

Sediment at each onsite DU and each background SU will be logged for lithologic characterization (color, moisture, texture, grain size, sorting, and lithology using USCS [ASTM 2006]). Information will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

ISM sediment sampling will be conducted during low tide when shoreline sediment is exposed. Prior to initiation of ISM sampling, the corners of onsite DUs and background SUs will be flagged. Location information for each onsite DU and background SU will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

Sediment increments will be collected from random locations within each onsite DU and each background SU using simple random sampling (ITRC 2020) in order to generate an unbiased estimate of the mean and variance. Each incremental sample will be comprised of 50 increments of sediment. Worksheet #11 presents the decision criteria used to determine the No. of increments comprising each incremental sample. Each of the triplicate incremental samples collected from onsite DUs (initial sample plus two additional samples) will be comprised of sediment increments taken from different, randomly selected locations within the DUs.

Sediment increments of equal volume will be collected using a dedicated incremental sampling tool with plungers collecting soil equivalent to approximately 33.3 g in volume, which will provide a total weight of 1 kg of sediment. The depth of each increment of sediment will be approximately 6 in. bgs, which is the length of the incremental sampling tool's sampling plunger attachment. Increments for each triplicate sample will not be collected from the same locations or co-located together.

Each incremental sample will be collected in a laboratory-approved bag or container large enough to accommodate the total 1-kg volume for analysis. ISM sediment samples will be submitted to Eurofins TestAmerica Canton for ISM processing. Metals and mercury samples will then be shipped to Eurofins TestAmerica Denver and TOC and grain size samples will be submitted to Katahdin Analytical Services, LLC, located in Scarborough, ME, for analysis.

The pH and ORP for ISM sediment samples will be determined in the field using pH and ORP meters. Approximately 20 g of well composited sediment will be mixed with 20 mL of deionized water and thoroughly mixed and homogenized. The suspension will then be allowed to rest to allow most of the suspended clay to settle out from the suspension prior to measuring pH and ORP. For ORP, both field and standard hydrogen electrode corrected values will be reported. pH and ORP will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

14.5.4 Discrete Sediment AVS/SEM Sampling Tasks

Discrete sediment AVS/SEM samples will be collected at low tide when shoreline sediment is exposed. Each sample will be collected from 0-6 in. bgs using dedicated sampling equipment (scoops/spoons) and/or clean/decontaminated, non-dedicated sampling equipment (hand augers or shovels). Any standing water that accumulated in the sample will be decanted/removed and sediment will be transferred to sample containers provided by the analytical laboratory.

Discrete samples will be collected for analysis AVS/SEM by SM 4500-S2D and EPA Method 6010C. AVS/SEM samples will be submitted to Eurofins Lancaster Laboratories, Inc., PA for offsite laboratory analysis.

Coordinates for sampling locations will be collected in the field with a hand-held GPS unit. Information will be documented in the field book in accordance with EA-SOP-016 (presented in Appendix H of the UFP-QAPP [EA 2020a]).

14.6 SAMPLE COLLECTION TASKS – EA

Prior to collecting field samples, samplers will don clean, protective gloves. Samples will be collected for constituents listed in Worksheet #18 of this UFP-QAPP Addendum. Sample containers will be provided by the analytical laboratory and will be pre-preserved for those constituents that require chemical preservation, as detailed in Worksheet #19 and #30 of this UFP-QAPP Addendum.

14.7 FIELD QUALITY CONTROL TASKS -EA

Field QC samples are intended to provide an indication of the consistency of sample collection and analyses over the course of the program. Field QC samples are listed on Worksheet #20 of this UFP-QAPP Addendum. Sampling locations are presented in Worksheet #18 of this UFP-QAPP Addendum.

ISM soil and sediment samples are being collected in triplicate (initial ISM sample plus two additional samples). Additional replicate samples are not required for ISM sediment samples. Duplicate discrete surface water samples will be collected at a rate of 1 per 20 samples, as required by the analytical method.

The analytical laboratory will perform MS/MSD on discrete surface water samples, and ISM soil and sediment samples at a rate of 1 per 20 samples, as required by the analytical method. Additional volume will be provided for MS/MSD.

14.8 SAMPLE HANDLING AND MANAGEMENT TASKS – EA

Samples will be placed in an iced cooler and maintained at less than 6 degrees Celsius (°C) (but not frozen) immediately upon collection. Samples will be labeled, packed, and shipped to the

analytical laboratory, and tracked by secure chain-of-custody protocol as detailed in Worksheet #26 and #27 of the UFP-QAPP (EA 2020a) and in accordance with EA-SOP-001 and EA-SOP-004 (presented in Appendix H of the UFP-QAPP [EA 2020a]). Records concerning the custody and condition of the samples will be maintained in field and laboratory records.

14.9 EQUIPMENT DECONTAMINATION TASKS – EA

Non-dedicated, reusable equipment that may directly or indirectly contact samples will be decontaminated between sampling locations in accordance with EA-SOP-005 (presented in Appendix H of the UFP-QAPP [EA 2020Aa). Reusable personal protective equipment or clothing that becomes contaminated during site work will be appropriately cleaned before reuse or will be disposed of and replaced.

14.10 INVESTIGATIVE DERIVED WASTE MANAGEMENT TASKS – EA

Expendable waste used in completing the sampling (including used personal protective equipment, dedicated disposable sampling equipment, and general trash) will be bagged and properly disposed of as general debris/trash. Decontamination wash water (deionized water and non-phosphate soap) will be limited and will be temporarily contained in 5-gallon buckets and discarded to the ground surface.

14.11 LABORATORY ANALYSIS TASKS – LABORATORY

The analytical laboratory will process and analyze field samples and field QC samples according to the sample chain-of-custody documents, and the requirements of the UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum. ISM soil and sediment samples will be processed by Eurofins TestAmerica Canton.

Each ISM sample will be air dried and disaggregated. Sample aliquots for grain size and CEC analysis will not be ground/milled as grinding would change sample grain size and adsorption capacity. Aliquots for TOC and mercury/metals analysis will be sieved and ground using the ball mill.

As was done during Phase I, grinding of metals/mercury samples will be conducted using a rolling ball mill with steel paint cans and ceramic cylinders (90% burundum) as grinding media. Milling durations will be consistent between samples, as conducted for Phase I samples and consistent with ITRC guidance (ITRC 2020).

Analytical parameter methods for each sample are presented in Worksheet #18 of this UFP-QAPP Addendum. Analytical SOPs are listed in Worksheet #23 of this UFP-QAPP Addendum. EPA screening values and laboratory reference limits for analytes are presented in Worksheet #15 of this UFP-QAPP Addendum.

14.12 LABORATORY QUALITY CONTROL TASKS – LABORATORY

Laboratory QC tasks provide an indication of accuracy, precision, and sensitivity of analytical methods for each analytical batch of field samples that are analyzed. The project laboratory will be responsible for conducting laboratory QC procedures and reporting laboratory QC results in accordance with laboratory SOPs listed in Worksheet #23 of this UFP-QAPP Addendum. The laboratory QA manager is responsible for ensuring that laboratory internal QC checks are conducted in accordance with applicable methods and protocols, the laboratory's QA manual and the requirements of UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum.

The laboratory QC samples are method specific, as detailed in Worksheet #12 and #28 of the UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum. Internal and continuing calibration verification (CCV) will be conducted for applicable equipment as summarized in Worksheet #24 and #25 of the UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum. Laboratory QC samples including ball mill grinding blanks, method blanks, laboratory control samples, MS/MSDs, and calibration check standards will be prepared and analyzed in accordance with the analytical method requirements and laboratory SOPs as listed in Worksheet #23 of the UFP-QAPP (EA 2020a) and this UFP-QAPP Addendum.

14.13 LABORATORY REPORTING TASKS – LABORATORY

Analytical data will be reviewed by the laboratory QC manager prior to providing the data to EA. The analytical laboratory will provide SEDD (Stage 2A) deliverables in accordance with the most recently published SEDD specification (e.g., Specification 5.2), as well as PDFs of the final analytical reports, including supporting documentation such as chromatograms and instrument calibrations. The laboratory will upload the SEDD files directly into the FUDSChem.

14.14 DATA VERIFICATION AND VALIDATION TASKS – EA

Review activities for analytical data and other project inputs are summarized in Worksheet #34 and #35 of the UFP-QAPP (EA 2020a). Data validation procedures for Phase II are presented in Worksheet #36 of this UFP-QAPP Addendum. Analytical data will be verified by the EA project chemist for completeness upon receipt. Corrective actions will be made upon decision of necessity to maintain the overall quality of the project data.

Once a SEDD is successfully uploaded and certified by TestAmerica in FUDSChem, an ADR process will be automatically initiated. The ADR will perform a Stage 2A data validation by comparing the SEDD to the approved eQAPP, summarizing QC outliers in an ADR Report, and applying data validation qualifiers to associated results. The "first review" EA chemist will perform a Stage 2B data validation based on the initial ADR Stage 2A validation. The data validator will review the ADR report against the PDF laboratory report to (1) verify/modify the ADR qualification of the sample results and to (2) supplement the ADR review with a manual review of QC elements that were not included in the ADR review. The data validator will complete the data review checklist and prepare a data validation report that summarizes the data validation

findings. The "second reviewer," EA chemist will review the data validation report and sign off as the second reviewer. The data validation report will then be uploaded to the FUDSChem library.

14.15 FUDSCHEM DATA MANAGEMENT – EA, LABORATORY

Analytical data will be managed in the FUDSChem library per the requirements specified in the USACE NAE Data Management Plan to ensure data deliverables follow Performance Work Statement data requirements (USACE NAE 2016). Other types of data that will be uploaded to FUDSChem include spatial information pertaining to ISM DU/SU, discrete surface water sampling locations, and discrete sediment AVS/SEM sample locations (northing, easting, and elevation). Shape files of the onsite approximately 1-acre (200 x 200 ft) soil DUs, ½-acre sized Iona Island sediment DUs and 0.15-acre sized Con Hook background sediment SUs will be provided to Synectics to be uploaded to FUDSChem.

14.16 DOCUMENTATION AND RECORDS TASKS – EA

A bound field logbook will be used to record information about each field activity as detailed in EA-SOP-059 (presented in Appendix H of the UFP-QAPP [EA 2020a]). Field activities will be recorded daily with black or blue waterproof ballpoint pens. Each page of field notes will be numbered and dated, and initials of crew members will be defined. Errors will be crossed out with a single line, initialed and dated, and correct data entered adjacent to the error. Pertinent information will be logged in the field logbook as follows:

- Personnel onsite and times onsite and offsite
- Site conditions (including presence of airborne particulates [soot, dust, etc.], and presence of unusual odors) and visitors onsite (as well as arrival and departure times) for each day are recorded
- The weather entry for each day including cloud cover (partly cloudy, full sun, etc.), precipitation (type and intensity), wind direction, temperature, wind speed, and humidity
- Daily tasks completed and the start/end time of tasks and references to applicable field sheets
- Sample collection information (location, matrix, sample identification No., date and time of sample collection, and analyses to be completed)
- Collection of QA/QC samples
- Field screening results
- References, such as maps or photographs of the sampling site.

The field logbooks will be maintained as follows:

- The cover of the field logbooks will list the project name, location, activities, name of contact and telephone No., start/end date, and time of logbook entries.
- No pages will be removed from the field logbooks.
- When author releases a specific field logbook, the new author will print his/her name, and will sign the field logbook prior to making any entries in the field logbook.

Field forms that will be completed for this project include health and safety forms provided in the APP (Appendix A of the UFP QAPP [EA 2020a]), chain-of-custody forms generated in FUDSChem, and water quality meter calibration logs provided in Appendix F of this UFP-QPP Addendum. Photographs will be used to document standard field procedures and conditions as well as unusual conditions observed during field activities. EA will gather documentation of field work and data collection and generation activities, review it for completeness, include it in reports as warranted, and place it in a central project repository.

14.17 ASSESSMENT/AUDIT TASKS – EA

SOPs will be reviewed prior to the performance of tasks. Assessment and oversight tasks are detailed in Worksheet #31, #32, and #33 of the UFP-QAPP (EA 2020a). Independent technical review and deliverable checks will be performed to assess the quality of field and reporting tasks. The Project Development Team will perform interdisciplinary checks to ensure minimal interference between tasks. The EA PM will be responsible for responding to the assessment findings, including corrective actions.

No external project-specific laboratory audits are planned by EA. The Laboratory QA manager will conduct assessments of the laboratory procedures and data as described in the laboratory's QA manual. The DoD-certified laboratories are periodically audited through the DoD ELAP as part of their accreditation process.

14.18 DATA EVALUATION TASKS – EA

A data package will be generated for this project by the analytical laboratory and will include a case narrative, chain-of-custody record, QC summary data, sample results, standards data, raw QC data, and bench sheets for each analytical method. The concentration measurements not known to be in error will be considered valid; suspect outliers will not be removed from the data set and will be included in the analyses. Concentrations below the method detection limit (MDL) will be reported as non-detect at the LOD with a U qualifier (e.g., if the LOD is 0.5 microgram per liter [µg/L], a concentration below the MDL will be set at 0.5 µg/L); a positive concentration detected below the LOQ but above the MDL will be at the concentration with a J qualifier (e.g., 0.2 µg/L J).

14.19 CALCULATION OF SEDIMENT EXPOSURE POINT CONCENTRATIONS -EA

The 95UCL of the data will be calculated for each approximately 1-acre (200 x 200 ft) soil DU and ½-acre sized Iona Island sediment DU based upon the results of the triplicate samples collected. The background 95UCL will be calculated for each Con Hook background sediment DU (Hudson River and Con Hook Marsh) based on the 9 background samples collected at 0.15-acre SUs established within each depositional environment. The Chebyshev 95UCL is selected to represent the EPC because it represents a more conservative estimate of the 95UCL than the Student t-Test 95UCL. The Chebyshev 95UCL will be calculated using the ITRC calculator (2020). If analytical results are rejected during data validation, the rejected results will be excluded from 95UCL calculations.

14.20 ISM SEDIMENT SAMPLES PERMUTATION TEST OF MEANS

Permutation test on means at the 95% confidence level was used during Phase I to compare Iona Island and background ISM soil analytical results. Permutation test on means at the 95% confidence level will be used for evaluating Phase II onsite versus background sediment results.

Permutation tests are a subset of nonparametric statistics. The two-sample permutation test of means is similar to a t-test in that it evaluates the difference between two group means, but in contrast to a t-test, the permutation test does not require that the data from each group follow a normal distribution or that the two groups have the same variance. For this reason, the permutation test is suitable for evaluating both small datasets and non-normal data distributions. Also, this permutation test does not require that the distribution of the two data groups have the same shape in order to be a central value test as does the commonly used Wilcoxon rank sum test.

The permutation test assumes only that the data from the two are "exchangeable" when H_0 is true. For a two-sample permutation test of means, the test statistic is the observed difference in the two group means (i.e., site versus background). If the H_0 is true, the group assignment is arbitrary, as there is no difference in the means and the data in essence come from the same population. Therefore, the data are randomly rearranged regardless of group assignment to produce a different set of group means in each rearrangement. The difference in group means is computed and stored after each rearrangement, and this process is repeated for either all possible rearrangements (if possible) or several thousand permutations for larger data sets. The proportion of differences from the rearrangements that equal or exceed the one observed difference from the original data is the permutation p-value of the test (Helsel et al. 2020).

Concentrations for constituents within each DU for sediment will be compared to background concentrations using a two-sample hypothesis test with the following H_0:

$$H_0\text{: Site} \leq = \text{Background}$$
$$H_a\text{: Site} > \text{Background}$$

14.21 RISK ASSESSMENT TASKS – EA

The Phase I preliminary HHRA and screening-level ecological risk assessment (SLERA) / refined screening-level ecological risk assessment (RSLERA) will be updated as needed to quantify exposures that result in potential risk. The risk assessments will follow various CERCLA guidance (e.g., EPA 1989, 1997, 1998, 2015) the USACE Risk Assessment Handbook, Volume I and II (USACE 2010), and the USACE NAE ERA Flowcharts. Specific details regarding the processes that will be followed for conducting the HHRA and ERA are presented in Appendix D and E the UFP-QAPP (EA 2020a). Additional detail for aquatic receptors is provided below. The HHRA and SLERA/RSLERA will be provided as appendices of the RI Report.

To evaluate potential risks to aquatic receptors, the following steps will be conducted:

For the SLERA:

1. Are the measured contaminants in surface water and sediment DoD-associated? For example, some metals are not associated with DoD activity (e.g., essential nutrients), and will be excluded from the risk assessment.

2. Are onsite sediment metals concentrations consistent with background? If statistical evaluations (permutation test on means at the 95% confidence level) indicate that site concentrations are similar to background, then the contaminant will be excluded from evaluation.

3. Do sediment 95UCL concentrations exceed conservative sediment screening values? If site concentrations exceed conservative EPA Region 4 sediment benchmarks (i.e., the Ecological Screening Value [ESV]) as presented in Appendix G, the contaminant will be considered a contaminant of potential ecological concern (COPEC), carried through to the RSLERA, and evaluated further. If site concentrations are lower than the conservative screening value, then potential risks are acceptable, the contaminant is not a COPEC, and no further evaluation is necessary. Note that for mercury and selenium, both of which are bioaccumulative, sediment 95UCLs will also be compared to screening values for aquatic life and for wildlife.

4. Discrete surface water concentrations will be compared to screening values for surface water (i.e., freshwater chronic values), adjusted for sample-specific hardness and dissolved concentrations, as necessary. Surface water screening values are presented in Worksheet #11. Concentrations exceeding screening values and background concentrations will be considered COPECs and carried forward to the RSLERA. In the RSLERA, dissolved metals concentrations will be used to evaluate potential risks to the aquatic community.

Selected sediment and surface water screening values are presented in Worksheet #11 and Appendix G of this UFP-QAPP Addendum.

For the RSLERA:

The RSLERA will examine the COPECs in light of less conservative, more site-specific and realistic assumptions consistent with refinements associated with Step 3a of EPA's 8-Step ERA Process and the decision process depicted on the NAE's RSLERA Flowchart. Assumptions to be refined include the following:

1. For lower-trophic level receptors (i.e., plants, benthic invertebrates, and aquatic life), less conservative sediment screening benchmarks/toxicity reference values will be used. These include EPA's Region 4 Refinement Screening Values for sediment. For surface water, differences between dissolved metals concentrations will be examined to evaluate potential impacts from particulates/suspended solids in the water column.

2. Exposure parameters for wildlife will reflect realistic, site-specific exposure and effects for the following proposed wildlife receptors:

   — Herbivorous mammals (muskrat)
   — Herbivorous birds (mallard duck)
   — Insectivorous birds (lesser scaup)
   — Piscivorous birds (great blue heron)
   — Piscivorous mammals (river otter).

   Exposure parameters for each receptor are presented in Appendix G of this UFP-QAPP Addendum.

3. To estimate tissue concentrations for the food web models various accumulation/uptake factors will be used. For plants, the bioaccumulation factors from EPA (2007) will be used. For sediment-dwelling invertebrates and benthic fish, biota-sediment accumulation factors from Bechtel Jacobs (1998) will be used. Proposed bioaccumulation factors and biota-sediment accumulation factors are provided in Appendix G of this UFP-QAPP Addendum.

   In the RSLERA, receptor-specific risks associated with both no observed adverse effect level (NOAEL) and lowest-observed-adverse-effect level (LOAEL) will be characterized and assessed with respect to population-level effects for the receptors. The SLERA process is conservative and is based on NOAELs. These represent the most conservative endpoint, in that no adverse effects were observed for the receptor at a given concentration or dose. When toxicological tests are performed the investigation looks for effects on growth or reproduction for a series of concentrations or doses. LOAELs are the lowest concentrations at which adverse effects have been observed on individual test organisms. The severity of effects considered low level varies based on the study from which LOAELs are derived; in general, they correspond to minor changes in growth or reproduction. LOAELs are useful because there is considerable uncertainty associated with NOAELs. Since NOAELs are associated with no observed effects in a test study, it is uncertain whether the values are close to or far below the threshold value at which effects would first be observed. LOAELs

thus serve to bound the range of NOAELs, and the threshold of toxic effects is considered to lie somewhere between the NOAEL and the LOAEL.

4. Therefore, LOAELs will be used as TRVs in the RSLERA. Proposed NOAELs and LOAELs are provided in the ERA Work Plan, presented as Appendix E of the UFP-QAPP (EA 2020a).

14.22 PHASE II REPORTING TASKS – EA

A TPP meeting will be held to present Phase II results to the government and stakeholders. An RI report will be prepared in accordance with CERCLA and will document the results of the Phase I and Phase II investigations and risk assessments. The RI report will present existing site conditions, the findings from the field efforts, an evaluation of the nature and extent of contamination, and the potential hazard or risk to human health and the environment. Preliminary Draft, Draft, Draft Final, and Final versions of the RI report will be prepared.

Data generated as part of the RI will be used to support the evaluation of remedial alternatives (as necessary) in a FS. If analytical results and risk assessments indicate that COPC/COPEC concentrations pose a potential risk to human health and/or the environment, then remedial alternatives will be developed to address those potential risks and to move forward through the remedial process toward respective future land use (i.e., conservation area) as a part of the FS. It should be noted that only unacceptable risks require remedial action. Only those specific constituents identified as COCs will be carried forward to the FS for development of remedial alternatives.

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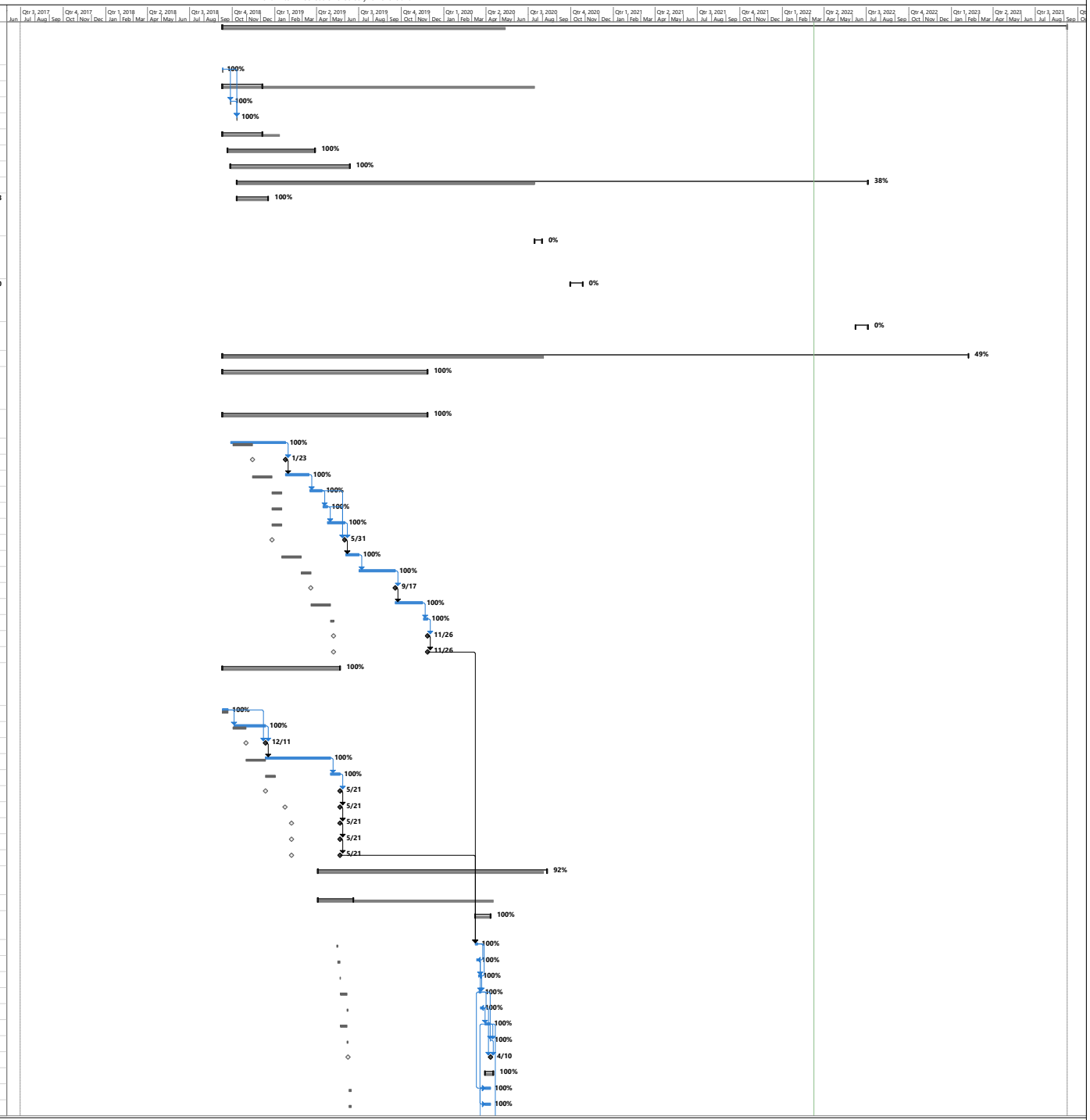
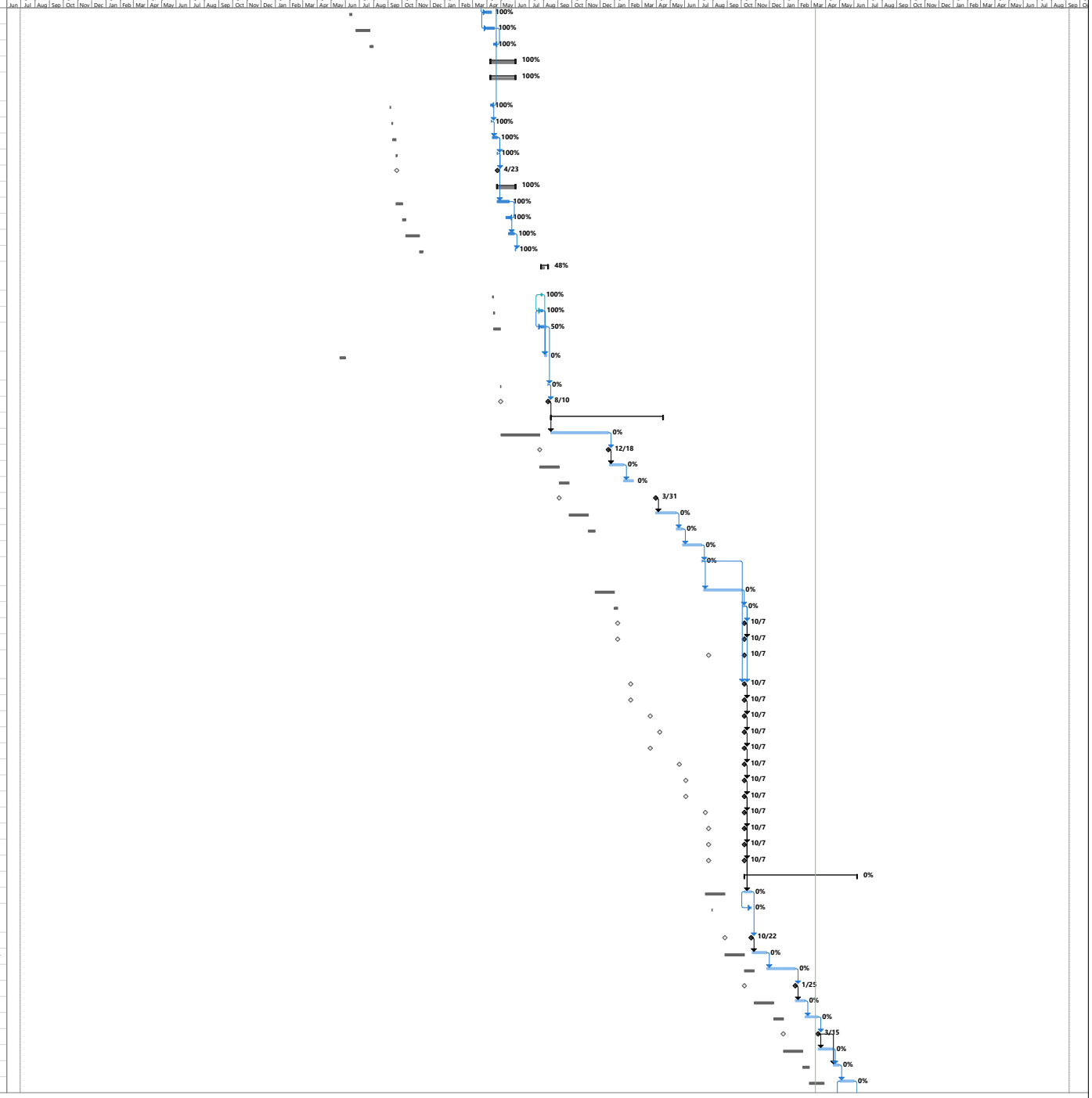
Iona RI to DD Schedule 28February for Iona Island

ID	Task Name	Duration	Start	Finish
1	**Iona Island FUDS - RI through DD at MRS-02 - MMRP - 1903 Explosion and 19 HTRW AOCs within 60 months of NTP (9/10/2018 - 9/09/2023)**	**1304 days?**	**Mon 9/10/18**	**Thu 9/7/23**
2	Notice to Proceed / Award (10 September 2018) - Iona Island FUDS	1 day	Mon 9/10/18	Mon 9/10/18
3	**CLIN 0001 Project Management Plan**	**62 days**	**Mon 9/10/18**	**Tue 12/4/18**
4	Kick-off Meeting with USACE and EA	1 day	Thu 9/27/18	Thu 9/27/18
5	Site Walk with USACE and Park Rangers	1 day	Thu 10/11/18	Thu 10/11/18
6	**CLIN 0001a - Project Management Plan (PMP)**	**62 days**	**Mon 9/10/18**	**Tue 12/4/18**
13	**CLIN 0001b Community Relations Plan (CRP)**	**135 days**	**Fri 9/21/18**	**Thu 3/28/19**
26	**Explosives Siting Plan (ESP) - To be completed by USACE**	**184 days**	**Thu 9/27/18**	**Tue 6/11/19**
36	**CLIN 0001c Meetings**	**973 days**	**Thu 10/11/18**	**Mon 7/4/22**
37	**CLIN 0001c-2 Technical Project Planning (TPP) Meeting #1 - Present MMRP and HTRW Approaches (Prior to Preliminary Draft WPs Submission to Project Delivery Team (PDT)**	**48 days**	**Thu 10/11/18**	**Mon 12/17/18**
47	**CLIN 0001c-3 TPP Meeting #2 - Present Digital Geophysical Mapping (DGM) Data and Initial HTRW Sample Results (after Fieldwork Session #1) to PDT**	**12 days**	**Wed 7/15/20**	**Thu 7/30/20**
54	**CLIN 0001c-4 TPP Meeting #3 - Present Intrusive Results and prior to Second Round of HTRW Sampling (before Fieldwork Session #2) PDT**	**19 days**	**Wed 9/30/20**	**Mon 10/26/20**
61	**CLIN 0001c-5 TPP Meeting #4 - Present MMRP Intrusive Results and follow-on HTRW Activities (after fieldwork Session #3) to PDT**	**19 days**	**Wed 6/8/22**	**Mon 7/4/22**
68	**CLIN 0002 MMRP MRS-1 1903 Explosion**	**1151 days?**	**Mon 9/10/18**	**Mon 2/6/23**
69	**CLIN 0002a-2 MMRP MRS-1 1903 Explosion - Remedial Investigation (RI) UFP (Uniform Federal Policy) - Quality Assurance Project Plan (QAPP) Work Plan (WP)**	**317 days**	**Mon 9/10/18**	**Tue 11/26/19**
70	**MMRP MRS-1 1903 Explosion - RI Work Plan (UFP-QAPP WP to include geophysical / MEC investigations)**	**317 days**	**Mon 9/10/18**	**Tue 11/26/19**
71	Preliminary Draft RI Work Plan Preparation	84 days	Fri 9/28/18	Wed 1/23/19
72	Submission of Preliminary Draft Remedial Investigation Work Plan	0 days	Wed 1/23/19	Wed 1/23/19
73	USACE Review	37 days	Thu 1/24/19	Fri 3/15/19
74	RLSO of Preliminary Draft RI Work Plan Preparation	20 days	Mon 3/18/19	Fri 4/12/19
75	USACE Back check RLSO Preliminary Draft RI Work Plan	8 days	Mon 4/15/19	Wed 4/24/19
76	Draft RI Work Plan Preparation	27 days	Thu 4/25/19	Fri 5/31/19
77	Submission of Draft RI Work Plan	0 days	Fri 5/31/19	Fri 5/31/19
78	Army Stakeholder Review of Draft RI Work Plan	21 days	Mon 6/3/19	Mon 7/1/19
79	Draft Final RI Work Plan Preparation	56 days	Tue 7/2/19	Tue 9/17/19
80	Submit Draft Final RI Work Plan	0 days	Tue 9/17/19	Tue 9/17/19
81	Draft Final RI Work Plan Review by NYSDEC and Property Owners	43 days	Wed 9/18/19	Fri 11/15/19
82	Final RI Work Plan Preparation	7 days	Mon 11/18/19	Tue 11/26/19
83	Submission of Final Remedial Investigation Work Plan	0 days	Tue 11/26/19	Tue 11/26/19
84	Final RI Work Plan Approval	0 days	Tue 11/26/19	Tue 11/26/19
85	**Accident Prevention Plan (APP) with Site Safety and Health Plan (SSHP) and Dive Plan (will ultimately be an appendix to both QAPPs - MMRP & HTRW)**	**182 days**	**Mon 9/10/18**	**Tue 5/21/19**
86	Develop Site Specific Hazard Evaluation for USACE Review	10 days	Mon 9/10/18	Fri 9/21/18
87	Preliminary Draft APP / SSHP Preparation	48 days	Fri 10/5/18	Tue 12/11/18
88	Submission of Preliminary Draft APP / SSHP	0 days	Tue 12/11/18	Tue 12/11/18
89	USACE Review	100 days	Wed 12/12/18	Tue 4/30/19
90	Draft APP / SSHP Preparation	15 days	Wed 5/1/19	Tue 5/21/19
91	Submission of Draft APP / SSHP	0 days	Tue 5/21/19	Tue 5/21/19
92	Army Stakeholder Review of Draft APP / SSHP	0 days	Tue 5/21/19	Tue 5/21/19
93	Final APP / SSHP Preparation	0 days	Tue 5/21/19	Tue 5/21/19
94	Submission of Final APP / SSHP	0 days	Tue 5/21/19	Tue 5/21/19
95	Final APP / SSHP Accepted	0 days	Tue 5/21/19	Tue 5/21/19
96	**CLIN 0002b MMRP MRS-1 1903 Explosion - Remedial Investigation Field Work**	**353 days**	**Thu 4/4/19**	**Mon 8/10/20**
97	**Field Session #1 - MMRP MRS-1 1903 Explosion**	**55 days**	**Thu 4/4/19**	**Wed 6/19/19**
98	**CLIN 0002b-1 Field Work Session #1 - MMRP MRS-1 1903 Explosion - Digital Geophysical Mapping (DGM)**	**25 days**	**Mon 3/9/20**	**Fri 4/10/20**
99	Mobilization, Site Setup, Brush Clearing, and Surface Clearance	5 days	Mon 3/9/20	Fri 3/13/20
100	DGM Mob in, Blind Seeding, etc.	2 days	Thu 3/12/20	Fri 3/13/20
101	DGM Instrument Verification Survey (IVS)	1 day	Mon 3/16/20	Mon 3/16/20
102	DGM Survey (Phase 1; prior to Covid-19 demobilization)	4 days	Tue 3/17/20	Fri 3/20/20
103	Demobilization DGM (for Covid-19)	1 day	Fri 3/20/20	Fri 3/20/20
104	DGM Survey (Phase 2)	9 days	Mon 3/30/20	Thu 4/9/20
105	Demobilization DGM (Following AGC)	1 day	Fri 4/10/20	Fri 4/10/20
106	Completion of DGM Field Work	0 days	Fri 4/10/20	Fri 4/10/20
107	**Data Processing #1 - DGM Data**	**14 days**	**Mon 3/30/20**	**Thu 4/16/20**
108	DGM Data Analysis	10 days	Mon 3/30/20	Fri 4/10/20
109	Target Anomaly Identification	10 days	Mon 3/30/20	Fri 4/10/20



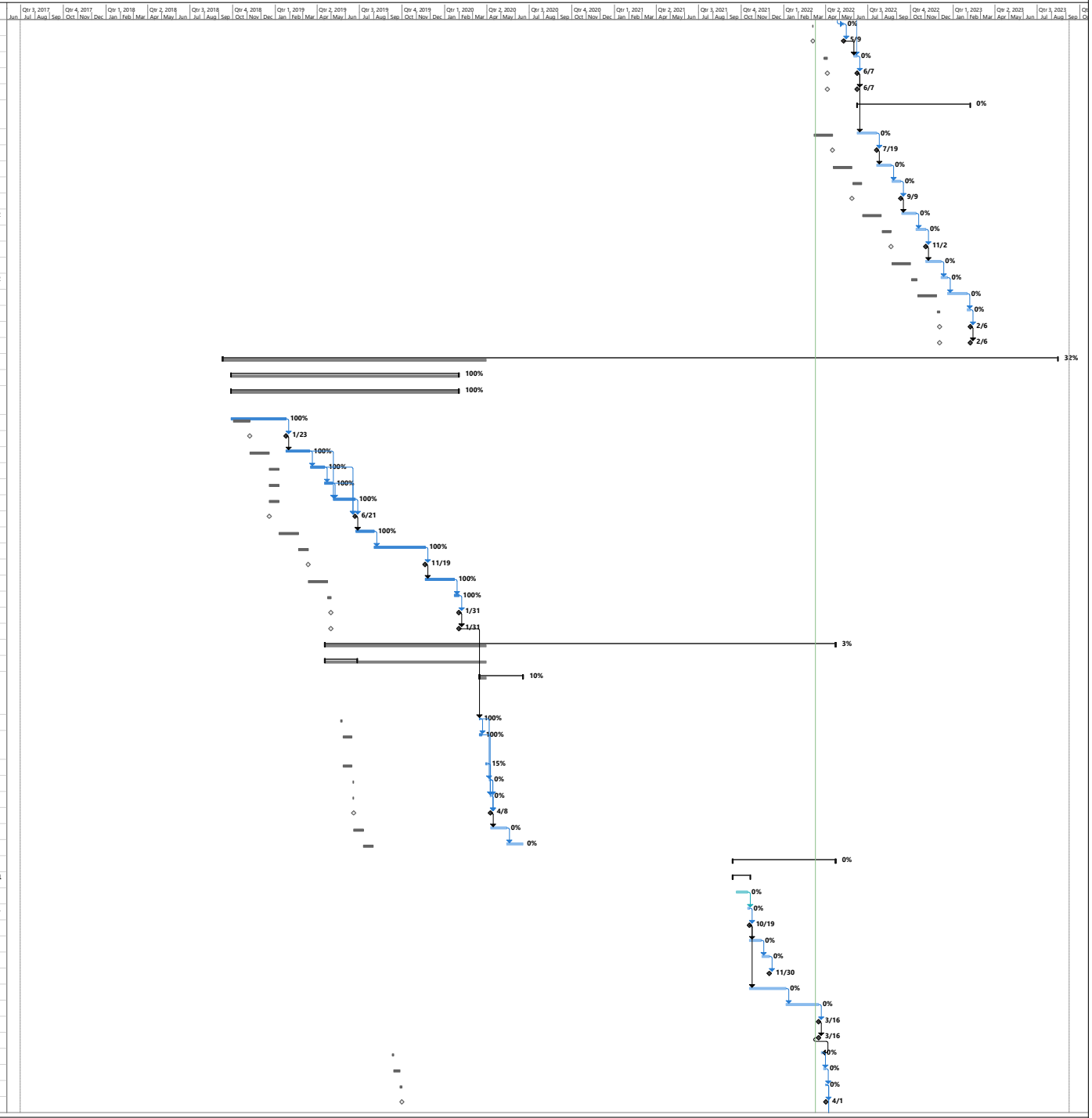
Critical | Critical Progress | Split | Manual Task | Finish-only | Baseline | Baseline Milestone | Summary Progress | Manual Summary | External Tasks | Inactive Task | Inactive Summary
Critical Split | Task | Task Progress | Start-only | Duration-only | Baseline Split | Milestone | Summary | Project Summary | External Milestone | Inactive Milestone | Deadline

ID	Task Name	Duration	Start	Finish
110	Potential Disposal Feature Identification	10 days	Mon 3/30/20	Fri 4/10/20
111	Review of Data with USACE (and Stakeholders if Requested)	12 days	Wed 4/1/20	Thu 4/16/20
112	Completion of Data Processing and Finalization of Cued List	1 day	Thu 4/16/20	Thu 4/16/20
113	**Field Session #2 - MMRP MRS-1 1903 Explosion**	**38 days**	**Thu 4/9/20**	**Mon 6/1/20**
114	**CLIN 0002b-2 Field Work Session #2 - MMRP MRS-1 1903 Explosion - Collect Cued Survey Data with AGC**	**38 days**	**Thu 4/9/20**	**Mon 6/1/20**
115	Mobilization, Site Setup, and Equipment Testing	1 day	Thu 4/9/20	Thu 4/9/20
116	AGC IVS	1 day	Fri 4/10/20	Fri 4/10/20
117	Reacquire and Cued Survey Anomalies with AGC	8 days	Mon 4/13/20	Wed 4/22/20
118	Demobilization	1 day	Thu 4/23/20	Thu 4/23/20
119	Completion of Field Work	0 days	Thu 4/23/20	Thu 4/23/20
120	**Data Processing #2 - AGC Data**	**28 days**	**Thu 4/23/20**	**Mon 6/1/20**
121	AGC Data Analysis	18 days	Thu 4/23/20	Mon 5/18/20
122	Generate "dig list" targets for Intrusive Investigation	5 days	Tue 5/12/20	Mon 5/18/20
123	Review of Data with USACE (and Stakeholders if Requested)	10 days	Mon 5/18/20	Fri 5/29/20
124	Completion of Data Processing	1 day	Mon 6/1/20	Mon 6/1/20
125	**CLIN 0002b-3 Field Work Session #3 - MMRP MRS-1 1903 Explosion - Intrusive Phase (Both Land and Water)**	**11 days**	**Mon 7/27/20**	**Mon 8/10/20**
126	Mobilization and Site Setup	2 days	Mon 7/27/20	Tue 7/28/20
127	Reacquisition of Selected AGC Anomalies	3 days	Mon 7/27/20	Wed 7/29/20
128	Intrusive Investigation of Selected AGC Anomalies as well as Shoreline, Dump Area, and Mag and Dig Anomalies	10 days	Mon 7/27/20	Fri 8/7/20
129	Dive on Three Loading Dock Areas, Video of Underwater Environment, and Intrusive Investigation of Marine Targets Encountered	5 days	Mon 8/3/20	Fri 8/7/20
130	Demobilization of Land and Dive UXO Teams	1 day	Mon 8/10/20	Mon 8/10/20
131	Completion of Field Work	0 days	Mon 8/10/20	Mon 8/10/20
132	**CLIN 0002c RI Report for MMRP MRS-01 1903 Explosion**	**174 days?**	**Mon 8/17/20**	**Thu 4/15/21**
133	Preliminary-Draft RI Report Preparation	90 days	Mon 8/17/20	Fri 12/18/20
134	Submission of Preliminary-Draft Remedial Investigation Report	0 days	Fri 12/18/20	Fri 12/18/20
135	USACE Review	23 days	Mon 12/21/20	Wed 1/20/21
136	Draft RI Report Preparation	15 days	Thu 1/21/21	Wed 2/10/21
137	Submission of Draft RI Report	0 days	Wed 3/31/21	Wed 3/31/21
138	Army Stakeholder Review of Draft RI Report	32 days	Wed 3/31/21	Thu 5/13/21
139	Draft Final RI Report Preparation	10 days	Fri 5/14/21	Thu 5/27/21
140	Army Stakeholder Backcheck	29 days	Fri 5/28/21	Wed 7/7/21
141	Submit Draft Final RI Report for Review by NYSDEC and Property Owners	2 days	Thu 7/8/21	Fri 7/9/21
142	Draft Final RI Report Review by NYSDEC and Property Owners	60 days	Mon 7/12/21	Fri 10/1/21
143	Final RI Report Preparation	4 days	Mon 10/4/21	Thu 10/7/21
144	Submission of Final Remedial Investigation Report (including EDD and GIS	0 days	Thu 10/7/21	Thu 10/7/21
145	Final RI Report Approval	0 days	Thu 10/7/21	Thu 10/7/21
146	**CLIN 0002d Feasibility Study (FS) for MMRP MRS-01 1903 Explosion (If Required)**	**0 days**	**Thu 10/7/21**	**Thu 10/7/21**
147	Preliminary Draft FS Preparation	0 days	Thu 10/7/21	Thu 10/7/21
148	Submission of Preliminary Draft Feasibility Study Report	0 days	Thu 10/7/21	Thu 10/7/21
149	USACE Review	0 days	Thu 10/7/21	Thu 10/7/21
150	Draft FS Report Preparation	0 days	Thu 10/7/21	Thu 10/7/21
151	Submission of Draft FS Report	0 days	Thu 10/7/21	Thu 10/7/21
152	Army Stakeholder Review of Draft FS Report	0 days	Thu 10/7/21	Thu 10/7/21
153	Draft Final FS Report Preparation	0 days	Thu 10/7/21	Thu 10/7/21
154	Submit Draft Final FS to NYSDEC & Property Owners Review	0 days	Thu 10/7/21	Thu 10/7/21
155	NYSDEC and Property Owners Review	0 days	Thu 10/7/21	Thu 10/7/21
156	Final FS Preparation	0 days	Thu 10/7/21	Thu 10/7/21
157	Submission of Final Feasibility Study Report	0 days	Thu 10/7/21	Thu 10/7/21
158	Final FS Approval	0 days	Thu 10/7/21	Thu 10/7/21
159	**CLIN 0002e Proposed Plan (PP) for MMRP MRS-01 1903 Explosion**	**173 days**	**Fri 10/8/21**	**Tue 6/7/22**
160	Preliminary Draft PP Preparation	11 days	Fri 10/8/21	Fri 10/22/21
161	Discuss (via teleconference) with Project Delivery Team Preliminary Draft of Proposed Plan	1 day	Fri 10/22/21	Fri 10/22/21
162	Submission of Preliminary Draft Proposed Plan	0 days	Fri 10/22/21	Fri 10/22/21
163	USACE Review	23 days	Mon 10/25/21	Wed 11/24/21
164	Draft PP Preparation	44 days	Thu 11/25/21	Tue 1/25/22
165	Submission of Draft PP	0 days	Tue 1/25/22	Tue 1/25/22
166	Army Stakeholder Review of Draft PP	15 days	Wed 1/26/22	Tue 2/15/22
167	Draft Final PP Preparation	20 days	Wed 2/16/22	Tue 3/15/22
168	Submit Draft Final PP to NYSDEC and Property Owners Review	0 days	Tue 3/15/22	Tue 3/15/22
169	NYSDEC and Property Owners Review	23 days	Wed 3/16/22	Fri 4/15/22
170	Incorporate NYSDEC & Property Owners Comments	10 days	Mon 4/18/22	Fri 4/29/22
171	Public Notice in Local Paper (30 Calendar Day Comment Period)	22 days	Mon 5/2/22	Tue 5/31/22



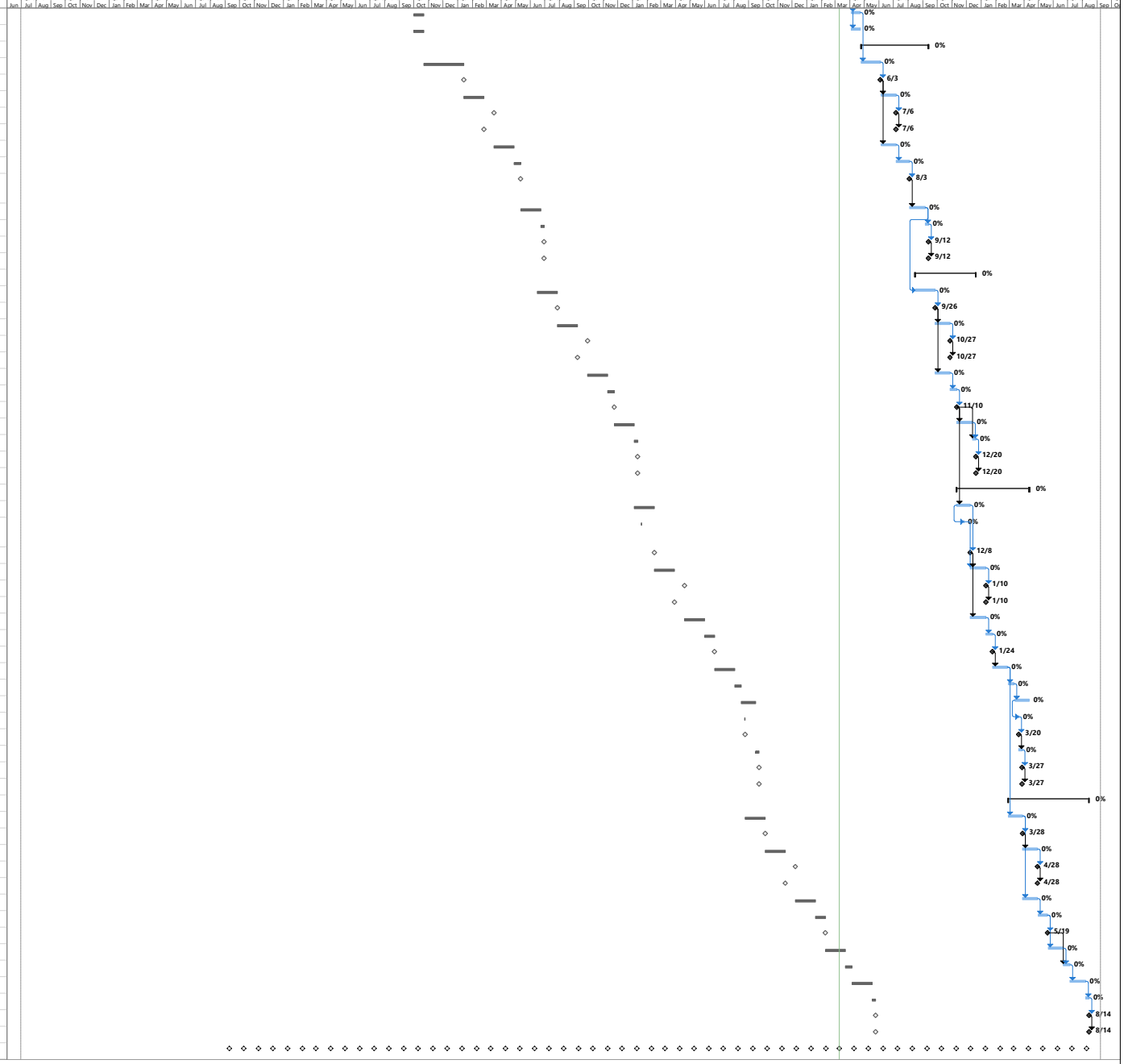
Critical | Critical Progress | Split | Manual Task | Finish-only | Baseline | Baseline Milestone | Summary Progress | Manual Summary | External Tasks | Inactive Task | Inactive Summary
Critical Split | Task | Task Progress | Start-only | Duration-only | Baseline Split | Milestone | Summary | Project Summary | External Milestone | Inactive Milestone | Deadline

ID	Task Name	Duration	Start	Finish
172	Public Meeting	1 day	Mon 5/9/22	Mon 5/9/22
173	Completion of Proposed Plan Public Meeting	0 days	Mon 5/9/22	Mon 5/9/22
174	Final PP Preparation	5 days	Wed 6/1/22	Tue 6/7/22
175	Submission of Final Proposed Plan	0 days	Tue 6/7/22	Tue 6/7/22
176	Final PP Approval	0 days	Tue 6/7/22	Tue 6/7/22
177	**CLIN 0002f Decision Document (DD) for MMRP MRS-01 1903 Explosion**	**174 days**	**Wed 6/8/22**	**Mon 2/6/23**
178	Preliminary Draft Decision Document Preparation	30 days	Wed 6/8/22	Tue 7/19/22
179	Submission of Preliminary Draft Decision Document	0 days	Tue 7/19/22	Tue 7/19/22
180	USACE Review	23 days	Wed 7/20/22	Fri 8/19/22
181	Draft Decision Document Preparation	15 days	Mon 8/22/22	Fri 9/9/22
182	Submission of Draft Decision Document	0 days	Fri 9/9/22	Fri 9/9/22
183	Army Stakeholder Review of Draft Decision Document	23 days	Mon 9/12/22	Wed 10/12/22
184	Draft Final Decision Document Preparation	15 days	Thu 10/13/22	Wed 11/2/22
185	Submit Draft Final DD to NYSDEC and Property Owners for Review	0 days	Wed 11/2/22	Wed 11/2/22
186	NYSDEC and Property Owners Review	23 days	Thu 11/3/22	Mon 12/5/22
187	Incorporate NYSDEC and Property Owners Comments	10 days	Tue 12/6/22	Mon 12/19/22
188	USACE HQ Review	30 days	Tue 12/20/22	Mon 1/30/23
189	Final Decision Document Preparation	5 days	Tue 1/31/23	Mon 2/6/23
190	Submission of Final Decision Document	0 days	Mon 2/6/23	Mon 2/6/23
191	Final Decision Document Approval	0 days	Mon 2/6/23	Mon 2/6/23
192	**CLIN 0003 HTRW AOCs**	**1286 days?**	**Mon 9/10/18**	**Mon 8/14/23**
193	**CLIN 0003a RI WP - HTRW AOCs**	**351 days**	**Fri 9/28/18**	**Fri 1/31/20**
194	**HTRW AOCS - RI Work Plan (HTRW investigations) (APP to be completed under MMRP WP Task)**	**351 days**	**Fri 9/28/18**	**Fri 1/31/20**
195	Preliminary Draft RI Work Plan Preparation	84 days	Fri 9/28/18	Wed 1/23/19
196	Submission of Preliminary Draft Remedial Investigation Work Plan	0 days	Wed 1/23/19	Wed 1/23/19
197	USACE Review	37 days	Thu 1/24/19	Fri 3/15/19
198	RLSO of Preliminary Draft Preparation	22 days	Mon 3/18/19	Tue 4/16/19
199	USACE Back check RLSO of Preliminary Draft RI Work Plan	13 days	Wed 4/17/19	Fri 5/3/19
200	Draft RI Work Plan Preparation (Including HHRA and Ecological Work Pla	35 days	Mon 5/6/19	Fri 6/21/19
201	Submission of Draft RI Work Plan	0 days	Fri 6/21/19	Fri 6/21/19
202	Army Stakeholder Review of Draft RI Work Plan	29 days	Mon 6/24/19	Thu 8/1/19
203	Draft Final RI Work Plan Preparation (includes CX back check)	78 days	Fri 8/2/19	Tue 11/19/19
204	Submit Draft Final RI Work Plan	0 days	Tue 11/19/19	Tue 11/19/19
205	Draft Final RI Work Plan Review by NYSDEC & Property Owners	45 days	Wed 11/20/19	Tue 1/21/20
206	Final RI Work Plan Preparation	8 days	Wed 1/22/20	Fri 1/31/20
207	Submission of Final Remedial Investigation Work Plan	0 days	Fri 1/31/20	Fri 1/31/20
208	Final RI Work Plan Approval	0 days	Fri 1/31/20	Fri 1/31/20
209	**CLIN 0003b Remedial Investigation Field Work - HTRW AOCs**	**787 days?**	**Thu 4/18/19**	**Fri 4/22/22**
210	**Field Session #1 - HTRW AOCs**	**50 days**	**Thu 4/18/19**	**Wed 6/26/19**
211	**CLIN 0003b-1 RI Fieldwork for Initial HTRW AOCs Sampling (Soil) for AOCs Not Previously Sampled or Nature and Extent of Characterized AOCs**	**68 days**	**Mon 3/16/20**	**Wed 6/17/20**
212	Mobilization & Site Setup	1 day	Mon 3/16/20	Mon 3/16/20
213	Field Sampling (Incremental Sampling) (Phase 1; prior to Covid-19 demobilization)	5 days	Mon 3/16/20	Fri 3/20/20
214	Field Sampling (Incremental Sampling) (Phase 2, After Remobilization	4 days	Mon 3/30/20	Thu 4/2/20
215	Shoreline Reconnaissance	2 days	Mon 4/6/20	Tue 4/7/20
216	Demobilization	1 day	Wed 4/8/20	Wed 4/8/20
217	Completion of IRP/HTRW Field Work	0 days	Wed 4/8/20	Wed 4/8/20
218	Data Analysis	25 days	Thu 4/9/20	Wed 5/13/20
219	Data Validation	25 days	Thu 5/14/20	Wed 6/17/20
220	**Field Session #2 - HTRW AOCs**	**160 days?**	**Mon 9/13/21**	**Fri 4/22/22**
221	**CLIN 0003b-2 RI Fieldwork for Data Gap Sampling of HTRW AOCs**	**28 days?**	**Mon 9/13/21**	**Wed 10/20/21**
222	USACE Back check RLSO of Preliminary Draft RI Work Plan	18 days	Tue 9/21/21	Thu 10/14/21
223	Draft RI Work Plan Preparation	2 days	Fri 10/15/21	Mon 10/18/21
224	Submission of Draft RI Work Plan	0 days	Tue 10/19/21	Tue 10/19/21
225	Army Stakeholder Review of Draft RI Work Plan	19 days	Tue 10/19/21	Fri 11/12/21
226	Draft Final RI Work Plan Preparation (includes CX back check)	12 days	Mon 11/15/21	Tue 11/30/21
227	Submit Draft Final RI Work Plan	0 days	Tue 11/30/21	Tue 11/30/21
228	Draft Final RI Work Plan Review by NYSDEC & Property Owners	57 days	Tue 10/19/21	Wed 1/5/22
229	Final RI Work Plan Preparation (includes back check)	50 days	Thu 1/6/22	Wed 3/16/22
230	Submission of Final Remedial Investigation Work Plan	0 days	Wed 3/16/22	Wed 3/16/22
231	Final RI Work Plan Approval	0 days	Wed 3/16/22	Wed 3/16/22
232	Mobilization and Site Setup	3 days	Wed 3/23/22	Fri 3/25/22
233	Field Sampling	4 days	Mon 3/28/22	Thu 3/31/22
234	Demobilization	1 day	Fri 4/1/22	Fri 4/1/22
235	Completion of Field Work	0 days	Fri 4/1/22	Fri 4/1/22



Critical | Critical Progress | Split | Manual Task | Finish-only | Baseline | Baseline Milestone | Summary Progress | Manual Summary | External Tasks | Inactive Task | Inactive Summary
Critical Split | Task | Task Progress | Start-only | Duration-only | Baseline Split | Milestone | Summary | Project Summary | External Milestone | Inactive Milestone | Deadline

ID	Task Name	Duration	Start	Finish
236	Data Analysis (2nd visit)	15 days	Mon 4/4/22	Fri 4/22/22
237	Data Validation (2nd Visit)	15 days	Mon 4/4/22	Fri 4/22/22
238	**CLIN 0003c RI Report for HTRW AOCs**	**101 days**	**Mon 4/25/22**	**Mon 9/12/22**
239	Preliminary-Draft RI Report Preparation	30 days	Mon 4/25/22	Fri 6/3/22
240	Submission of Preliminary-Draft Remedial Investigation Report	0 days	Fri 6/3/22	Fri 6/3/22
241	USACE Review	23 days	Mon 6/6/22	Wed 7/6/22
242	Draft RI Report Preparation	0 days	Wed 7/6/22	Wed 7/6/22
243	Submission of Draft RI Report	0 days	Wed 7/6/22	Wed 7/6/22
244	Army Stakeholder Review of Draft RI Report	23 days	Mon 6/6/22	Wed 7/6/22
245	Draft Final RI Report Preparation	20 days	Thu 7/7/22	Wed 8/3/22
246	Submit Draft Final RI Report for Review by NYSDEC & Property Owners	0 days	Wed 8/3/22	Wed 8/3/22
247	Draft Final RI Report Review by NYSDEC & Property Owner's	23 days	Thu 8/4/22	Mon 9/5/22
248	Final RI Report Preparation	5 days	Tue 9/6/22	Mon 9/12/22
249	Submission of Final Remedial Investigation Report	0 days	Mon 9/12/22	Mon 9/12/22
250	Final RI Report Approval (including EDD and GIS Data)	0 days	Mon 9/12/22	Mon 9/12/22
251	**CLIN 0003d FS for HTRW AOCs**	**91 days**	**Tue 8/16/22**	**Tue 12/20/22**
252	Preliminary Draft FS Preparation	30 days	Tue 8/16/22	Mon 9/26/22
253	Submission of Preliminary Draft Feasibility Study Report	0 days	Mon 9/26/22	Mon 9/26/22
254	USACE Review	23 days	Tue 9/27/22	Thu 10/27/22
255	Draft FS Report Preparation	0 days	Thu 10/27/22	Thu 10/27/22
256	Submission of Draft FS Report	0 days	Thu 10/27/22	Thu 10/27/22
257	Army Stakeholder Review of Draft FS Report	23 days	Tue 9/27/22	Thu 10/27/22
258	Draft Final FS Report Preparation	10 days	Fri 10/28/22	Thu 11/10/22
259	Submit Draft Final FS to NYSDEC & Property Owners Review	0 days	Thu 11/10/22	Thu 11/10/22
260	NYSDEC & Property Owners Review	23 days	Fri 11/11/22	Tue 12/13/22
261	Final FS Preparation	5 days	Wed 12/14/22	Tue 12/20/22
262	Submission of Final Feasibility Study Report	0 days	Tue 12/20/22	Tue 12/20/22
263	Final FS Approval	0 days	Tue 12/20/22	Tue 12/20/22
264	**CLIN 0003e - PP for HTRW AOCs**	**108 days**	**Fri 11/11/22**	**Tue 4/11/23**
265	Preliminary Draft PP Preparation	20 days	Fri 11/11/22	Thu 12/8/22
266	Discuss (via teleconference) with Project Delivery Team Preliminary Draft of Proposed Plan	1 day	Fri 11/25/22	Fri 11/25/22
267	Submission of Preliminary Draft Proposed Plan	0 days	Thu 12/8/22	Thu 12/8/22
268	USACE Review	23 days	Fri 12/9/22	Tue 1/10/23
269	Draft PP Preparation	0 days	Tue 1/10/23	Tue 1/10/23
270	Submission of Draft PP	0 days	Tue 1/10/23	Tue 1/10/23
271	Army Stakeholder Review of Draft PP	23 days	Fri 12/9/22	Tue 1/10/23
272	Draft Final PP Preparation	10 days	Wed 1/11/23	Tue 1/24/23
273	Submit Draft Final PP to NYSDEC & Property Owners Review	0 days	Tue 1/24/23	Tue 1/24/23
274	NYSDEC & Property Owners Review	23 days	Wed 1/25/23	Fri 2/24/23
275	Incorporate NYSDEC & Property Owners Comments	10 days	Mon 2/27/23	Fri 3/10/23
276	Public Notice in Local Paper (30 Calendar Day Comment Period)	22 days	Mon 3/13/23	Tue 4/11/23
277	Public Meeting	1 day	Mon 3/20/23	Mon 3/20/23
278	Completion of Proposed Plan Public Meeting	0 days	Mon 3/20/23	Mon 3/20/23
279	Final PP Preparation	5 days	Tue 3/21/23	Mon 3/27/23
280	Submission of Final Proposed Plan	0 days	Mon 3/27/23	Mon 3/27/23
281	Final PP Approval	0 days	Mon 3/27/23	Mon 3/27/23
282	**CLIN 0003f DD for HTRW AOCs**	**121 days**	**Mon 2/27/23**	**Mon 8/14/23**
283	Preliminary Draft Decision Document Preparation	22 days	Mon 2/27/23	Tue 3/28/23
284	Submission of Preliminary Draft Decision Document	0 days	Tue 3/28/23	Tue 3/28/23
285	USACE Review	23 days	Wed 3/29/23	Fri 4/28/23
286	Draft Decision Document Preparation	0 days	Fri 4/28/23	Fri 4/28/23
287	Submission of Draft Decision Document	0 days	Fri 4/28/23	Fri 4/28/23
288	Army Stakeholder Review of Draft Decision Document	23 days	Wed 3/29/23	Fri 4/28/23
289	Draft Final Decision Document Preparation	15 days	Mon 5/1/23	Fri 5/19/23
290	Submit Draft Final DD to NYSDEC & Property Owners for Review	0 days	Fri 5/19/23	Fri 5/19/23
291	NYSDEC & Property Owners Review	23 days	Mon 5/22/23	Wed 6/21/23
292	Incorporate NYSDEC & Property Owners Comments	10 days	Thu 6/22/23	Wed 7/5/23
293	USACE HQ Review	23 days	Thu 7/6/23	Mon 8/7/23
294	Final Decision Document Preparation	5 days	Tue 8/8/23	Mon 8/14/23
295	Submission of Final Decision Document	0 days	Mon 8/14/23	Mon 8/14/23
296	Final Decision Document Approval	0 days	Mon 8/14/23	Mon 8/14/23
297	**Monthly Status Report (Completed when DDs are Finalized)**	**1283 days**	**Mon 9/10/18**	**Thu 8/10/23**



Critical | Critical Progress | Split | Manual Task | Finish-only | Baseline | Baseline Milestone | Summary Progress | Manual Summary | External Tasks | Inactive Task | Inactive Summary
Critical Split | Task | Task Progress | Start-only | Duration-only | Baseline Split | Milestone | Summary | Project Summary | External Milestone | Inactive Milestone | Deadline

UFP-QAPP ADDENDUM WORKSHEET #15 – PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

This worksheet presents analytical methods, analytes, project screening values, and achievable laboratory limits including LOQs, LODs, and MDLs. The purpose of this worksheet is to determine if laboratory limits will meet EPA screening values for human health and ecological receptors. The reference limit and evaluation tables in this worksheet and in Worksheet #15 of the UFP-QAPP (EA 2020a) present EPA screening values for human health and ecological receptors, and achievable laboratory limits. Analytes without screening values will be reported for consideration in the risk assessment. If analytes without established risk-based screening values are detected, these analytes will be assessed using alternative means such as use of surrogate compounds.

EPA screening values for surface water samples are as follows:

- Human health screening values: EPA MCLs and tapwater RSLs (most recent version at the time of report preparation). The lesser of the two values will be prioritized for use in the HHRA.

- Ecological screening values: The default screening value was obtained from the NRWQC for aquatic life, chronic freshwater (available: https://www.epa.gov/wqc/national-recommended-water-quality-criteria-aquatic-life-criteria-table). For analytes without NRWQC, additional values were selected from EPA Regions 3 or 4. Refer to Worksheet #11.

EPA screening values for sediment samples are as follows:

- Human health screening values: EPA residential and industrial RSLs for soil (most recent version at the time of report preparation). Note: Industrial RSLs will be used for comparison purposes only. For the HHRA, residential soil RSL be used to determine COPCs in sediment.

- Ecological screening values: Due to a lack of EPA sediment screening values for Region 2 (which includes New York), freshwater sediment screening values from surrounding EPA regions were evaluated to determine appropriate screening values for sediment. Region 3 (2006) and Region 4 (2018) have ecological screening values for sediment, and values for Region 4 are either identical to or lower than those of Region 3. Therefore, Region 4 ESV are the most applicable for the site. Values are presented in Worksheet #15 and Appendix G.

Concentrations exceeding applicable EPA screening values will be compared to background concentrations obtained from Con Hook (using statistical analyses) to determine whether concentrations are consistent with or exceed background. Contaminants with concentrations greater than both screening values and respective background concentrations will be identified as

COPCs and/or COPECs and will be further evaluated in the HHRA and/or ERA. Further detail on conducting the HHRA and ERA, including target risk thresholds, is presented in Section 14.21 of this QAPP Addendum and in the risk assessment work plans presented as Appendix D of the UFP-QAPP (EA 2020a).

The MDL is the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99 percent level of confidence. Although a result at or above the MDL indicates that the analyte is present, the absence of a result at or above the MDL is inconclusive (i.e., one cannot confidently state whether the analyte is present or absent), because the false negative rate at the MDL is 50 percent. The MDL is used to determine the LOD for each analyte and matrix as well as for preparatory and cleanup methods routinely used on samples.

The LOD is the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99 percent confidence level. If a sample has a true concentration at the LOD, there is a minimum probability of 99 percent of reporting a detection (a measured value greater than or equal to the MDL) and a 1 percent chance of reporting a non-detect (a false negative). Due to the false negative rate at the LOD (1percent), the laboratory will report non-detected values as less than the LOD (i.e., the LOD will be reported with a U qualifier).

The LOQ is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. Quantitative concentration results within specified limits of precision and bias can only be achieved at or above the LOQ; however, the analytical laboratory may identify analytes between the MDL and the LOQ. In these instances, the laboratory will report concentration values between the MDL and the LOQ as estimated (J) values.

To meet MQOs for sensitivity, the LODs for non-detects and LOQs for detects need to be below the project action levels (PALs). PALs have been set at the lowest screening value for each analyte. When MQOs for sensitivity are not met, the results will not be usable for determining whether the analyte concentrations are above or below the PALs. The most sensitive commercially available analytical methods are being utilized for the analyses presented in this UFP-QAPP Addendum. However, there are circumstances where theoretically determined toxicity values are so low that commercially available analytical methods cannot achieve them.

In cases where the LOQ is less than one third the PAL, the project quantitation limit (PQL) goal is set at one-third the PAL, in order to maximize the likelihood that the MQOs are met, in accordance with UFP-QAPP guidance. Where the LOQ is greater than one-third the PAL, the PQL goal is set to the LOQ, for consistency with the analytical quantitation limit. If there is no PAL, then the PQL goal is set to five times the LOQ.

Analytes with LOQs and LODs that are higher than the screening values are presented below. EPA screening values in red in the following tables indicate where the laboratory LOD is higher than the screening value. Cells highlighted in yellow indicate where the laboratory LOQ is higher than the screening values.

- The LOQ and LOD for surface water analysis of antimony, arsenic, cadmium, and thallium by EPA SW 846 Method 6020A and mercury by EPA SW 846 7470A exceed the EPA Tapwater RSLs . In addition, the LOQ exceeds the RSL for cobalt.

- The LOQ and LOD for surface water analysis of cadmium by EPA SW 846 Method 6020A exceed EPA's Ecological Screening Level (freshwater chronic). In addition, the LOQ exceeds the screening values for aluminum, lead, and silver.

- The LOQ for sediment analysis of thallium by EPA SW 846 Method 6020A exceeds the residential soil RSL.

When the PAL is less than the LOD and/or LOQ, LODs for non-detects and J-qualified detected results less than the LOQ are not usable for reliably determining whether contaminant concentrations are above or below the PALs. The project team will work closely with the laboratory prior to evaluation to try to reduce the reporting limits to meet the PALs. This may be achieved by increasing the amount of sample (e.g., surface water) collected and/or by alternative preparation methods to reduce any required dilutions due to matrix interferences.

The path forward for all analytes that have LOQs that exceed screening values will be discussed by the PDT once the analytical data are received and prior to the risk evaluation. Any results that are qualified as non-detect (e.g., U-qualified) will be evaluated on an individual basis for data trends, site relevance, background concentration, etc. Any values that are J-qualified with LOQs greater than screening levels will also be reviewed for potential quality issues, site relevance, and data trends.

For surface water human health risk, antimony, arsenic, cadmium, mercury, and thallium have LODs that exceed screening-values for tapwater. These tapwater screening values are typically used to evaluate groundwater as a drinking water source, so they are extremely conservative. It should be noted that the MDLs for surface water meet all screening values except for the RSL for arsenic and thallium. Since arsenic and thallium are unlikely to be major risk drivers, uncertainties associated with non-detect data are likely to be minimal. If arsenic and/or thallium concentrations are U-qualified, the concentrations will be examined for data trends, site relevance, background concentration, etc. Although there is uncertainty with these data, if arsenic and thallium are not anticipated to be risk drivers or site-related (i.e., not detected in soil and/or sediment at elevated levels), arsenic and thallium concentrations in surface water will not be considered significant.

For ecological risk in surface water, only cadmium has an LOD that is slightly greater (1.0) than the ecological screening value (0.72). If cadmium is U-qualified, the uncertainty section will discuss the fact that the MDL is sufficiently low, and concentrations will be examined for data trends, site relevance, background concentration, etc. Although there is uncertainty with these data, if cadmium is not anticipated to be a risk driver or site-related (i.e., not detected in soil and/or sediment at elevated levels), cadmium concentrations in surface water will not be considered a risk-driver.

Matrix effects or necessary dilutions may affect the laboratory limits reported for project samples. A discussion will be presented in the uncertainty sections of the risk assessments.

The following table presents the location of reference limit and evaluation tables for each Phase II media and laboratory analysis. The following table presents the location of reference limit and evaluation tables for each Phase II media and laboratory analysis. Note: PALs and achievable laboratory limits for TOC analysis in soil and sediment by Walkley-Black are presented in the UFP-QAPP (EA 2020a). There are no PALs or laboratory limits for grain size by ASTM D422.

Surface Water Analyses	
TAL Metals – Method 6010C and 6020A	Table 15-1 of this UFP-QAPP Addendum
Mercury –Method 7470A	Table 15-1 of this UFP-QAPP Addendum
Hardness – SM2340B	NA
Sediment Analyses	
Metals – Method 6010C and 6020A	Table 15-2 of this UFP-QAPP Addendum
Mercury – Method SW7471	Table 15-2 of this UFP-QAPP Addendum
Sulfide (for AVS/SEM) by SM 4500-S2D-2011	Table 15-3 of this UFP-QAPP Addendum
Metals – ICP Water (for AVS/SEM) by SW-846 6010C	Table 15-3 of this UFP-QAPP Addendum
Total Organic Carbon –Method Walkley-Black	Table 15-6 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	NA
Soil Analyses	
Total Organic Carbon –Method Walkley-Black	Table 15-6 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	NA
Cation-Exchange Capacity SW-846 Method 9081	Table 15-4 of this UFP-QAPP Addendum

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
ICP = Inductively coupled plasma
NA = Not applicable
SEM = Simultaneously extracted metals
SM = Standard method
TAL = Target Analyte List

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Table 15-1 – Reference Limits and Evaluation Table for Metals – Water (Eurofins TestAmerica Denver)

Matrix: Water
Analytical Group: TAL Metals by EPA SW 846 Methods 6010C/6020A and Mercury by EPA SW Method 7470A
Concentration Level: Low

Analyte	CAS No.	EPA Screening Values[1]			Project Action Limit (µg/L)[5]	PQL Goal (µg/L)[6]	Achievable Laboratory Limits (µg/L)		
		EPA Tapwater RSLs (µg/L)[2]	EPA MCLs (µg/L)[3]	Ecological Screening Levels Freshwater Chronic (µg/L)[4]			MDL	LOD	LOQ
Aluminum	7429-90-5	2,000	NS	87	87	300	18	70	300
Antimony	7440-36-0	**0.78**	6.0	190	0.78	6.0	0.40	1.0	6.0
Arsenic	7440-38-2	**0.052**	10	150	0.052	5.0	0.33	1.0	5.0
Barium	7440-39-3	380	2,000	220	220	73	0.29	0.95	3.0
Beryllium	7440-41-7	2.5	4.0	11	2.5	1.0	0.080	0.30	1.0
Cadmium	7440-43-9	**0.92**	5.0	**0.72**	0.72	1.0	0.265	1.0	1.0
Calcium	7440-70-2	NS	NS	116,000	116,000	38,700	77.8	160	1,000
Chromium, Total	7440-47-3	2,200	NS	74	74	14	0.50	1.8	10
Cobalt	7440-48-4	0.60	NS	19	0.60	1.0	0.0923	0.35	1.0
Copper	7440-50-8	80	1,300	4.95	4.95	2.0	0.56	1.8	2.0
Iron	7439-89-6	1,400	NS	1,000	1,000	333	22	85	100
Lead	7439-92-1	15	15	2.5	2.5	3.0	0.18	0.70	3.0
Magnesium	7439-95-4	NS	NS	82,000	82,000	27,333	26.4	60	500
Manganese	7439-96-5	43	NS	93	43	14	0.31	0.95	3.5
Mercury	7439-97-6	**0.063**	2.0	0.77	0.063	0.20	0.027	0.080	0.20
Nickel	7440-02-0	39	NS	52	39	9.6	0.30	1.0	3.0
Potassium	7440-09-7	NS	NS	53,000	53,000	15,000	237	940	3,000
Selenium	7782-49-2	10	50	5.0	5.0	5.0	0.373	1.0	5.0
Silver	7440-22-4	9.4	NS	1.6	1.6	5.0	0.033	0.10	5.0
Sodium	7440-23-5	NS	NS	680,000	680,000	227,000	373	1,000	5,000
Thallium	7440-28-0	**0.020**	2.0	6.0	0.020	1.0	0.089	0.20	1.0
Vanadium	7440-62-2	8.6	NS	27	8.6	6.0	1.2	3.0	6.0
Zinc	7440-66-6	600	NS	120	120	22	2.0	8.0	20

1) Highlighted cells indicate screening values that are lower than the laboratory LOQ. Red values indicate screening values that are lower than the laboratory LOD.
2) EPA Regional Screening Levels Table, EPA, May 2021. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
3) EPA Maximum Contaminant Levels.
4) Refer to ecological screening value selection, provided in Worksheet #11.
5) Project action limit is the lowest of all available screening values.
6) The PQL Goal is set to 1/3 of the project action limit. If the LOQ is greater than 1/3 the project action limit, then the PQL goal is set to the LOQ. If there are no screening values, then the PQL goal is set to 5 times the LOQ.

NOTES:
CAS = Chemical abstracts service
EPA = U.S. Environmental Protection Agency
HI = Hazard index
LOD = Limit of detection
LOQ = Limit of quantitation
MDL = Method detection limit
µg/L = Milligram(s) per liter
NS = No standard
PQL = Project quantitation limit RSL = Regional screening level
TAL = Target Analyte List

Table 15-2 – Reference Limits and Evaluation Table for Metals – Sediment (Eurofins TestAmerica Denver)

Matrix: Sediment
Analytical Group: Metals by EPA SW 846 Methods 6010C/6020A and Mercury by EPA SW Method 7471B
Concentration Level: Low

Analyte	CAS No.	EPA Screening Values[1]				Project Action Limit (mg/kg)[4]	PQL Goal (mg/kg)[5]	Achievable Laboratory Limits (mg/kg)		
		Residential Soil RSL (mg/kg)[2]	Industrial Soil RSL (mg/kg)[2]	EPA Region 4 Ecological Screening Levels Freshwater Sediment ESV (mg/kg)[3]	Source			MDL	LOD	LOQ
Aluminum	7429-90-5	7,700	110,000	25,000	LANL	7,700	2,567	1.55	6.0	50
Antimony	7440-36-0	3.1	47	2.0	Long and Morgan, 1991	2.0	0.70	0.0819	0.10	0.20
Arsenic	7440-38-2	0.68	3.0	9.8	MacDonald et al. 2003	0.68	0.60	0.0506	0.20	0.60
Barium	7440-39-3	1,500	22,000	20	MacDonald et al. 2003	20	6.7	0.0705	0.20	0.25
Beryllium	7440-41-7	16	230	NS	-	16	5.3	0.0225	0.080	0.10
Cadmium	7440-43-9	7.1	98	1.0	MacDonald et al. 2003	1.0	0.30	0.00938	0.035	0.10
Chromium, Total	7440-47-3	NS	NS	43.4	MacDonald et al. 2003	43.4	14.5	0.076	0.175	0.60
Cobalt	7440-48-4	12,000	180,000	50	MacDonald et al. 2003	50	17	0.00663	0.025	0.10
Copper	7440-50-8	310	4,700	31.6	MacDonald et al. 2003	31.6	10.5	0.0711	0.20	2.5
Iron	7439-89-6	5,500	82,000	20,000	Persaud et al. 1993	5,500	1,833	8.27	20	80
Lead	7439-92-1	400	800	35.8	MacDonald et al. 2003	35.8	11.9	0.0182	0.070	0.40
Manganese	7439-96-5	180	2,600	460	Persaud et al. 1993	180	60	0.0961	0.30	1.0
Mercury	7439-97-6	1.1	4.6	0.18	MacDonald et al. 2003	0.18	0.060	0.00553	0.0133	0.017
Nickel	7440-02-0	150	2,200	22.7	MacDonald et al. 2003	22.7	7.57	0.169	0.35	0.35
Selenium	7782-49-2	39	580	0.72	LANL	0.72	0.50	0.133	0.40	0.50
Silver	7440-22-4	39	580	1.0	MacDonald et al. 2003	1.0	0.30	0.00783	0.025	0.10
Thallium	7440-28-0	0.078	1.2	NS	-	0.078	0.10	0.00351	0.010	0.10
Vanadium	7440-62-2	39	580	NS	-	39	13	0.104	0.30	0.50
Zinc	7440-66-6	2,300	35,000	121	MacDonald et al. 2003	121	40.3	0.688	1.5	2.5

1) Highlighted cells indicate screening levels that are lower than the laboratory LOQ.
2) EPA Regional Screening Levels Table, EPA, May 2021. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
3) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018.
4) The project action limit is the lowest of the EPA screening values presented.
5) The PQL Goal is set to 1/3 of the project action limit. If the LOQ is greater than 1/3 the project action limit, then the PQL goal is set to the LOQ. If there are no screening values, then the PQL goal is set to 5 times the LOQ.

NOTES:
CAS = Chemical abstracts service
EPA = U.S. Environmental Protection Agency
HI = Hazard index
LANL = Los Alamos National Laboratory
LOD = Limit of detection
LOQ = Limit of quantitation
MDL = Method detection limit
mg/kg = Milligram(s) per kilogram
NS = No standard
PQL = Project quantitation limit
RSL = Regional screening level
TAL = Target Analyte List

Table 15-3 – Reference Limits and Evaluation Table for Sulfide and Metals (for AVS/SEM) – Sediment (Eurofins Lancaster Laboratories, Inc.)

Matrix: Sediment AVS/SEM extract
Analytical Group: Sulfide by SM 4500-S2D-2011 and Metals by SW-846 6010C
Concentration Level: Low

Analyte	CAS No.	PQL Goal (µmol/g)[1]	Achievable Laboratory Limits (µmol/g)		
			MDL	LOD	LOQ
AVS	18496-25-8	3.15	0.200	0.400	0.630
Cadmium	7440-43-9	0.02225	0.000890	0.00356	0.00445
Copper	7440-50-8	0.1575	0.0121	0.0299	0.0315
Lead	7439-92-1	0.0362	0.00290	0.00676	0.00724
Nickel	7440-02-0	0.085	0.00443	0.0153	0.0170
Zinc	7440-66-6	0.153	0.0153	0.0291	0.0306

1) There are no screening values for AVS/SEM. The PQL goal is set to 5 times the LOQ.

NOTES:
AVS = Acid volatile sulfide
CAS = Chemical abstracts service
HI = Hazard index
LOD = Limit of detection
LOQ = Limit of quantitation
MDL = Method detection limit
PQL = Project quantitation limit
SEM = Simultaneously extracted metals
SM = Standard method
µmol/g = Micromoles per gram

Table 15-4 – Reference Limits and Evaluation Table for Cation-Exchange Capacity – Soil (Eurofins TestAmerica Corpus Christi)

Matrix: Soil Extract
Analytical Group: Cation-Exchange Capacity by SW-846 Method 9081 and Method 6010B
Concentration Level: Low

Analyte	CAS No.	PQL Goal (mg/L)[1]	Achievable Laboratory Limits[2] (mg/L)	
			MDL	RL
Sodium	7440-23-5	5	0.3100	1.0000

1) There are no screening values for CEC. The PQL goal is set to 5 times the RL.
2) Eurofins TestAmerica, Corpus Christi is not DoD accredited, however the laboratory has been deemed acceptable by the USACE project chemist for CEC analysis. Laboratory limits are presented as MDL and RL.

NOTES:
CAS = Chemical Abstracts Service
CEC = Cation exchange capacity
DoD = Department of Defense
MDL = Method detection limit
RL = Reporting limit
mg/L = Milligram(s) per liter

UFP-QAPP ADDENDUM WORKSHEET #17 – SAMPLE DESIGN AND RATIONALE

This worksheet documents the overall process for the design and rationale of the sampling program for Phase II. This worksheet is used to develop and document the investigative approach, in terms of the types of activities/procedures to be conducted, investigative locations, field methodologies, matrices to be sampled, analytical groups to be analyzed and at what concentration levels (QC samples), the No. of samples to be taken, and the sampling frequency (including seasonal considerations).

If the field conditions encountered during Phase II warrant changes to the field tasks or sampling plans, the Field Team Leader will notify the EA PM immediately upon discovery. Field changes will be communicated as presented in Worksheet #6 of this UFP-QAPP Addendum. Specifically, once notified, the EA PM will notify the USACE PM and USACE TM within 24 hours verbally or via email. Based on a review of the proposed change, and if required by the USACE, a field change request memorandum will be submitted within 1 week to the USACE PM and USACE TM for review and approval. It should be noted that unanticipated field changes may require a UFP-QAPP Addendum, amendment, and/or revision. This requirement will be determined in consultation with the USACE PM and USACE TM following notification of the proposed change. Any UFP-QAPP Addendum, amendment, and/or revision will be submitted to the USACE and regulators for review, comment, and approval.

17.1 DESCRIBE AND PROVIDE A RATIONALE FOR CHOOSING THE SAMPLING APPROACH

Phase II is being conducted to close data gaps identified after completion of Phase I and support decision making, including:

- Evaluate bioavailability of metals in onsite upland and hydric soil to support development of risk assessments.

- Evaluate the potential for offsite migration of metals from soil at AOC #16 – Area D (Dumping Area) to the shoreline environment via the surface water pathways identified during Phase I (including tidal flow to the Hudson River and migration along the riprap filled drainage swale and tidal wetland swale to Iona Marsh).

- Evaluate whether metals associated with AOC #16 – Area D (Dumping Area) are present in Hudson River and Iona Marsh shoreline sediment at concentrations that may pose risk to human health and/or ecological receptors. An ISM approach has been selected for shoreline sediment sampling to reduce data variability and increase sample representativeness, consistent with Phase I.

- Evaluate bioavailability of metals in sediment to support development of risk assessments.

- Assess the potential for toxicity to sediment-dwelling organisms to support development of the ERA.

Phase II media to be investigated, sample locations, depths, times, and analyses have been selected to evaluate migration pathways to the shoreline environment, impacts to environmental media, and potential for risk to human health and/or ecological receptors. Phase II sampling data needs and sampling rationale are presented in Tables 17-1 and 17-2. A summary of Phase II field activities is presented in the subsections below. Sampling locations will be adjusted, if needed, based on conditions encountered during field activities.

17.1.1 Iona Island Bioavailability Parameter Upland and Hydric Soil Sampling

Onsite ISM soil sampling will be conducted to evaluate physical parameters (TOC, grain size, CEC, and ORP) to assess the bioavailability of detected metals and PAHs in Phase I upland and hydric soil samples and support development of the ERA. DU-4, DU-7, DU-9, DU-11, and DU-13 are targeted for physical parameter sampling during Phase II to evaluate bioavailability in upland soil. DU-17, DU-18, and DU-19 are targeted for physical parameter sampling during Phase II to evaluate bioavailability in hydric soil. Physical parameter samples from each DU will be collected in triplicate (initial ISM sample plus two additional samples) to allow calculation of the 95UCL for each analytical parameter (Figure 11-1).

Upland and hydric soil samples will be analyzed for the following parameters (Table 17-1 and Worksheet #18):

- Grain size by ASTM D422
- TOC by Walkley Black
- CEC by EPA SW-846 Method 9081
- ORP by field screening (both field and standard hydrogen electrode corrected values).

17.1.2 AOC #16 – Area D (former Dumping Area) Surface Water Sampling

For discrete sampling, sample sizes of at least 20 are typically needed to compare mean site concentrations with mean background concentrations using hypothesis tests for centrality. Therefore, 20 samples will be collected from each onsite area targeted during Phase II, for a total of 60 discrete surface water grab samples to evaluate migration of metals from AOC #16 – Area D (former Dumping Area) to the shoreline via surface water pathways identified during Phase I (Figure 11-2a):

- 20 samples within AOC #16 – Area D (former Dumping Area) during tidal inundation (rising or high tide) to evaluate mobilization and transport of contaminants from soil during tidal inundation (DUMP-SW-01 to DUMP-SW-20)

- 20 samples along the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area) during high or receding tide to evaluate metals transported to the river shoreline (IHRS-SW-01 to IHRS-SW-20)

- 20 samples along the Iona Marsh shoreline at the outlets of the riprap filled drainage swale and tidal wetland swale (10 samples at each outlet location) to evaluate metals transported to the marsh along the swales (RRDS-SW-01 to RRDS-SW-10 and TWS-SW-01 to TWS-SW-10).

Surface water will be submitted for offsite laboratory analysis of the following parameters (Table 17-1 and Worksheet #18):

- Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A
- Dissolved (field filtered) mercury by EPA Method 7470A
- Hardness by SM2340B.

For completeness purposes, the full list of TAL metals is being evaluated for surface water samples. However, those metals that are not a CERCLA hazardous substance (for example, manganese) will be ineligible for DERP FUDS response.

Field screening of surface water at each sampling location will be conducted for temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity to compare background representativeness and support the risk assessments.

17.1.3 Con Hook Background Surface Water Sampling

Discrete background surface water grab samples will be collected within each background SU to establish background concentrations of TAL metals (including mercury) in surface water. For discrete sampling, sample sizes of at least 20 are typically needed to compare mean site concentrations with mean background concentrations using hypothesis tests for centrality. Therefore, 20 samples will be collected along the Hudson River shoreline (CHHRS-SW-01 through CHHRS-SW-20) and twenty samples will be collected along the Con Hook Marsh shoreline (CHMS-SW-01 through CHMS-SW-20) (Figure 11-3). Background surface water will be collected during low tide concurrent with sediment sampling and submitted for offsite laboratory analysis of the following parameters (Table 17-1 and Worksheet #18):

- Dissolved TAL (field filtered) metals by EPA Methods 6010C/6020A
- Dissolved (field filtered) mercury by EPA Method 7470A
- Hardness by SM2340B.

Field screening of surface water at each sampling location will be conducted for temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity to compare background representativeness.

17.1.4 AOC #16 – Area D (former Dumping Area) ISM Sediment Sampling

Three DUs, each sized at approximately ½-acre, have been established at Iona Island, with one DU along the Hudson River shoreline and two DUs along the Iona Marsh shoreline to evaluate concentrations in sediment associated with AOC #16 – Area D (former Dumping Area) as detailed in Worksheet #11 (Figure 11-2b). In addition, samples will be collected for physical parameter data (grain size, TOC, pH, and ORP) to evaluate bioavailability of metals (including mercury) and support development of the RSLERA.

- IHRS-DU-01: Established along the Hudson River shoreline along the eastern boundary of AOC #16 – Area D (former Dumping Area) to evaluate whether metals from with AOC #16 – Area D (former Dumping Area) have accumulated in Hudson River shoreline sediment

- RRDS-DU-01: Established along the Iona Marsh shoreline at the outlet of the riprap filled drainage swale to evaluate whether metals (including mercury) from AOC #16 – Area D (former Dumping Area) have been transported via the swale to the marsh shoreline and accumulated in sediment

- TWS-DU-01: Established along the Iona Marsh shoreline at the outlet of the tidal wetland swale to evaluate whether metals from AOC #16 – Area D (former Dumping Area) have been transported via the swale to the marsh shoreline and accumulated in sediment.

DUs are sized at ½-acre as this represents the smallest area for which ecological receptors may be exposed. ISM sediment sampling will be conducted in triplicate (initial ISM sample plus two additional samples) at each Iona Island sediment DU (9 samples total) to calculate the 95UCL for each analytical parameter. ISM sediment samples will be collected at low tide when shoreline sediment is exposed, and samples will be analyzed for the following parameters (Table 17-1 and Worksheet #18):

- Metals by EPA Methods 6010C/6020A
- Mercury by EPA Method 7471B
- TOC by Walkley Black
- Grain size by ASTM D422
- pH by field screening
- ORP by field screening (both field and standard hydrogen electrode corrected values)

17.1.5 AOC #16 – Area D (former Dumping Area) Discrete Sediment AVS/SEM Sampling

Discrete sediment samples will be collected from the Hudson River and Iona Marsh shorelines for analysis of AVS/SEM to assess the potential for toxicity to sediment-dwelling organisms in each within each onsite Phase II sediment DU and support development of the ERA. Samples will be collected at 0-6 in. bgs at the locations presented in Figure 11-2b. Three discrete sediment samples

will be collected along the Hudson River shoreline within IHRS-DU-01 (IHRS-SED-01 to IHRS-SED-03). Six discrete sediment samples will be collected along the Iona Marsh shoreline, including 3 within RRDS-DU-01 (RRDS-SED-01 to RRDS-SED-03) and 3 within TWS-DU-01 (TWS-SED-01 to TWS-SED-03).

Discrete sediment samples will be collected at low tide when shoreline sediment is exposed and analyzed for AVS/SEM by SM 4500-S2D and EPA Method 6010C (Table 17-1 and Worksheet #18).

17.1.6 Con Hook Background ISM Sediment Sampling

Two background DUs have been established at Con Hook, one on the Hudson River shoreline and one on the Con Hook Marsh shoreline, to evaluate background metals concentrations in sediment within each depositional environment. In addition, physical parameter data (TOC, grain size, pH, and ORP) will also be collected from each DU to evaluate background representativeness.

Each DU will consist of eight 0.15-acre SUs, with eight SUs on the Hudson River Shoreline (CHHR-SU-01 through CHHR-SU-08) and eight 0.15-acre SUs along the Con Hook Marsh shoreline (CHMS-SU-01 through CHMS-SU-08) (Figure 11-3). Due to spatial constraints along the shoreline at Con Hook, each SU was sized at 0.15 acres (162 x 40.5 ft along the Hudson River shoreline and 81 ft x 81 ft along the marsh shoreline). Single ISM samples will be collected from each 0.15-acre background sediment SU. Background ISM sediment samples will be collected at low tide when shoreline sediment is exposed and analyzed for the following parameters (Table 17-1 and Worksheet #18):

- Metals by EPA Methods 6010C/6020A
- Mercury by EPA Method 7471B
- TOC by Walkley Black
- Grain size by ASTM D422
- pH by field screening
- ORP by field screening (both field and standard hydrogen electrode corrected values)

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Table 17-1 Phase II Data Needs

Sample Matrix	Parameter	Analytical Method	Rationale for Analysis	Data Use
ISM soil samples	TOC	Walkley-Black	Additional data is needed to evaluate bioavailability of metals and PAHs to terrestrial receptors exposed to upland and hydric soil.	Data will be used to provide additional lines of evidence for assessing bioavailability in onsite soil both within individual DUs and across the Iona Island and to support development of the ERA.
	Grain size	ASTM D422		
	CEC	EPA SW-846 Method 9081 and Method 6010B		
	ORP	Field screening using an ORP meter (both field and standard hydrogen electrode corrected values)		
Surface Water Samples				
Discrete surface water samples	Dissolved (field filtered) TAL metals	EPA Methods 6010C/6020A	TAL metals (including mercury) data are needed to evaluate the potential for migration of contaminants from soil to the shoreline via surface water pathways. AOC #16 – Area D (former Dumping Area) is inundated during high tide and storm events. Contaminants in soil may be mobilized during tidal inundation and transported to the Hudson River shoreline during high or receding tide. In addition, contaminants may be transported to Iona Marsh along drainage swales from the western boundary of AOC #16 – Area D (former Dumping Area) (riprap filled drainage swale and tidal wetland swale).	TAL metals (including mercury) data will be evaluated to determine if contaminants are migrating from AOC #16 – Area D (former Dumping Area) to the Hudson River and Iona Marsh shorelines at concentrations above both at EPA screening values and background concentrations, and if concentrations may pose risk to human health and/or ecological receptors. Upstream background surface water sampling data will be used to establish regional background/ upstream metals concentrations outside the influence of Iona Island.
	Dissolved (field filtered) mercury	EPA Method 7470A		
	Hardness	EPA Method SM 2340B		EPA water screening values for metals are hardness dependent. Hardness data will be used to calculate site-specific surface water screening values for metals and compare background representativeness.
	Temperature	Field screening using a water quality meter		General water quality parameter data will be used to compare background representativeness and support the risk assessments.
	pH			
	Specific conductivity			
	DO			
	ORP			
	Turbidity			
	TSS			
	Salinity			
Sediment Samples				
ISM sediment samples	Metals	EPA Methods 6010C/6020A	Additional data is needed to determine whether metals (including mercury) related to AOC #16 – Area D (former Dumping Area) have accumulated in shoreline sediment at concentrations that pose risk to human and ecological receptors. An ISM approach has been selected for shoreline sediment sampling to reduce data variability and increase sample representativeness, consistent with Phase I.	Metals sediment data will be used to determine if contaminants associated with AOC #16 – Area D (former Dumping Area) are present in sediment along the Hudson River and Iona Marsh shorelines at concentrations above both EPA screening values and background concentrations, and if concentrations may pose risk to human health and/or ecological receptors. Upstream background sediment sampling data will be used to establish regional background/upstream metals concentrations outside the influence of Iona Island.
	Mercury	EPA Method 7471B		
	TOC	Walkley-Black	Additional data is needed to determine whether metals (including mercury) in sediment are bioavailable to ecological receptors. As AVS/SEM samples cannot be collected via ISM, bioavailability samples will be collected using discrete sampling methods.	TOC data will be used to describe sediment habitat type and provide additional lines of evidence for assessing the toxicity of sediment and bioavailability of metals compare background representativeness and support the risk assessments.
	Grain size	ASTM D422		Grain size data will be used to describe sediment conditions at sampling locations, qualitatively evaluate fate and transport processes based on patterns of deposition and sediment grain size distribution, provide additional lines of evidence for assessing the bioavailability of metals compare background representativeness, and support the risk assessments.
	pH	Field screening using a pH meter		pH data will be used to provide additional lines of evidence for assessing the toxicity of sediment and bioavailability of metals compare background representativeness and support the risk assessments.
	ORP	Field screening using an ORP meter (both field and standard hydrogen electrode corrected values)		ORP data will be used to provide additional lines of evidence for assessing the toxicity of sediment and bioavailability of metals compare background representativeness and support the risk assessments.
Discrete Sediment Samples	AVS/SEM	SM 4500-S2D and EPA Method 6010C	Additional data is needed to assess the potential for toxicity to sediment-dwelling organisms within each onsite Phase II sediment DU.	AVS/SEM data will be used to determine the potential for metal ions found in sediment to cause toxic effects in organisms dwelling in the sediment and to support the ecological risk assessment.

NOTES:
95UCL = 95th percent upper confidence limit of the mean
AOC = Area of concern
ASTM = ASTM International
AVS = Acid volatile sulfide
bgs = Below ground surface
CEC = Cation-exchange capacity
DO = Dissolved oxygen
DU = Decision unit
EPA = U.S. Environmental Protection Agency
ERA = Ecological risk assessment
in. = Inch(es)
ISM = Incremental sampling methodology
ORP = Oxidation-reduction potential
PAH = Polycyclic aromatic hydrocarbon
SEM = Simultaneously extracted metals
SIM = Selected ion monitoring
SM = Standard method
TAL = Target Analyte List
TOC = Total organic carbon
TSS = Total suspended solids

Table 17-2 Sampling Rationale

Sample Location/ Decision Unit[1]	Rationale for Sampling	Primary AOC Investigated
Onsite Soil Samples		
DU-4 established during Phase I	To evaluate bioavailability of metals and PAHs in onsite soil. DUs were selected to provide reasonable spatial coverage/representation across Iona Island. From a topographic and physical features point-of-view, the physical parameters within the 8 DUs are representative of all conditions at Iona Island. Therefore, physical parameters from the 8 DUs will be used to conduct a sitewide evaluation of bioavailability parameters, as well as to conduct an evaluation on a DU-by-DU basis. Three ISM soil samples will be collected from each Iona Island sediment DU consistent with Phase I pH data to account for potential variability in each DU and generate a Chebyshev 95UCL for each parameter within each DU. In addition, a 95UCL for each parameter will be generated for the entirety of Iona Island to evaluate bioavailability in DUs that are not being sampled in Phase II. Each ISM soil sample will consist of 50 increments of sediment, which is sufficient to estimate the reasonable unbiased estimate of concentrations across each approximately 1-acre (200 ft x 200 ft) sized DU.	Eight onsite DUs established during Phase I, including 5 DUs within terrestrial upland surface soil of Iona Island and 3 DUs within marsh hydric soil between Iona and Round Islands
DU-7 established during Phase I		
DU-9 established during Phase I		
DU-11 established during Phase I		
DU-13 established during Phase I		
DU-17 established during Phase I		
DU-18 established during Phase I		
DU-19 established during Phase I		
Iona Island Surface Water Samples		
AOC #16 – Area D (former Dumping Area) (DUMP-SW-01 to DUMP-SW-20)	To evaluate whether contaminants previously identified in soil at AOC #16 – Area D (former Dumping Area) (i.e., metals) are being mobilized during tidal inundation at concentrations that may pose risk to human health and/or ecological receptors.	AOC #16 – Area D (former Dumping Area)
Hudson River shoreline along eastern boundary of AOC #16 – Area D (former Dumping Area) (IHRS-SW-01 to IHRS-SW-20)	To evaluate whether contaminants previously identified in soil at AOC #16 – Area D (former Dumping Area) (i.e., metals) are being transported to the Hudson River shoreline during tidal fluctuations at concentrations that may pose risk to human health and/or ecological receptors.	
Iona Marsh shoreline at outlet of riprap filled drainage swale (RRDS-SW-01 to RRDS-SW-10)	To evaluate whether contaminants previously identified in soil at AOC #16 – Area D (former Dumping Area) (i.e., metals) are being transported to Iona Marsh via drainage pathways identified during Phase I leading from the western boundary of AOC #16 – Area D (former Dumping Area) to the marsh (i.e., riprap filled drainage swale and the tidal wetland swale) at concentrations that may pose risk to human health and/or ecological receptors.	
Iona Marsh shoreline at outlet of tidal wetland swale (TWS-SW-01 to TWS-SW-10)		
Background Surface Water Samples		
Con Hook Hudson River Shoreline (CHHRS-SW-01 to CHHRS-SW-20)	To establish regional surface water background concentrations of metals in Hudson River depositional environment. Analytical data will be used in background comparison for surface water samples.	Background samples collected from Con Hook along Hudson River shoreline
Con Hook Marsh shoreline (CHMS-SW-01 to CHMS-SW-20)	To establish regional background concentrations of metals in marsh depositional environment. Analytical data will be used in background comparison for surface water samples.	Background samples collected from Con Hook marsh shoreline
Iona Island ISM Sediment Samples		
Hudson River shoreline (IHRS-DU-01), sized at ½-acre	To evaluate whether contaminants previously identified in soil at AOC #16 – Area D (former Dumping Area) (i.e., metals) have accumulated in sediment along the Hudson River and Iona Marsh shorelines at concentrations that may pose risk to human health and/or ecological receptors; to evaluate bioavailability of metals in sediment; and to support the risk assessments. DUs were established at the ends of preferential flow pathways. Samples will be collected in triplicate to calculate the 95UCL using statistical tests. Each ISM sediment sample will consist of 50 increments of sediment, which is sufficient to estimate the reasonable unbiased estimate of concentrations across the ½-acre DU. Iona Island sediment DUs are sized at ½-acre as this represents the smallest area for which ecological receptors may be exposed.	AOC #16 – Area D (former Dumping Area)
Riprap filled drainage swale outlet at Iona Marsh (RRDS-DU-01), sized at ½-acre		
Tidal wetland swale outlet at Iona Marsh (TWS-DU-01), sized at ½-acre		
Background ISM Sediment Samples		
Con Hook Hudson River shoreline DU comprised of 8 SUs (CHHRS-SU-01 to CHHRS-SU-08), each SUsized at 0.15 acre	To determine background concentrations of metals in Hudson River sediment and evaluate background representativeness. Analytical data will be used in background comparison for sediment samples. Samples will be collected in triplicate to provide background dataset of 9 samples and calculate the 95UCL for the background Hudson River DU using statistical tests. Each ISM sediment sample will consist of 50 increments of sediment, which is sufficient to estimate the reasonable unbiased estimate of concentrations across each ½-acre SU. SUs were sized at ½-acre to be consistent with Iona Island sediment DU sizes.	Background samples collected from Con Hook along Hudson River shoreline
Con Hook marsh mhoreline DU comprised of 8 SUs CHMS-SU-01 to CHMS-SU-08), each SU sized at 0.15 acre		Background samples collected along Con Hook marsh shoreline

Sample Location/ Decision Unit[1]	Rationale for Sampling	Primary AOC Investigated
Iona Island Discrete Sediment AVS/SEM Samples		
Hudson River shoreline	To provide an indicator of metals bioavailability and potential to cause toxicity and to support the risk assessments. Three samples will be collected from each Iona Island sediment DU to account for potential variability in each DU.	AOC #16 – Area D (former Dumping Area)
Riprap filled drainage swale outlet at Iona Marsh		
Tidal wetland swale outlet at Iona Marsh		

NOTES:
95UCL = 95th percent upper confidence limit of the mean
AOC = Area of concern
DU – Decision unit
ERA = Ecological risk assessment
HMW = High molecular weight
ISM = Incremental sampling methodology
LMW = Low molecular weight
NA = Not applicable
PAH = Polycyclic aromatic hydrocarbon
SU = Sampling unit
TAL = Target Analyte List

UFP-QAPP ADDENDUM WORKSHEET #18 – SAMPLING LOCATIONS AND METHODS

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Iona Island ISM Surface Soil Samples									
DU-4	ISM	3	900589.41	636714.34	0–6 in. bgs	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-057	TOC by Walkley Black Grain size by ASTM D422 CEC by EPA SW-846 Method 9081 ORP by field screening (both field and standard hydrogen electrode corrected values)	IINY-DU-04A-SSIS-YYYYMMDD IINY-DU-04B-SSIS-YYYYMMDD IINY-DU-04C -SSIS-YYYYMMDD	None
			900774.36	636638.45					
			900850.25	636823.41					
			900665.29	636899.29					
DU-7	ISM	3	900088.86	636123.89	0–6 in. bgs			IINY-DU-07A-SSIS-YYYYMMDD IINY-DU-07B-SSIS-YYYYMMDD IINY-DU-07C -SSIS-YYYYMMDD	MS/MSD (extra volume collected) for TOC and CEC from 1 of 3 samples only. MS/MSD for grain size and ORP samples are NA.
			900273.81	636048.00					
			900349.73	636232.95					
			900164.78	636308.84					
DU-9	ISM	3	899429.97	636116.35	0–6 in. bgs			IINY-DU-09A-SSIS-YYYYMMDD IINY-DU-09B-SSIS-YYYYMMDD IINY-DU-09C -SSIS-YYYYMMDD	None
			899613.40	636195.87					
			899533.88	636379.26					
			899350.53	636299.74					
DU-11	ISM	3	899853.62	636390.49	0–6 in. bgs			IINY-DU-11A-SSIS-YYYYMMDD IINY-DU-11B-SSIS-YYYYMMDD IINY-DU-11C -SSIS-YYYYMMDD	None
			900038.57	636314.57					
			900114.45	636499.52					
			899929.50	636575.40					
DU-13	ISM	3	899540.99	636678.03	0–6 in. bgs			IINY-DU-13A-SSIS-YYYYMMDD IINY-DU-13B-SSIS-YYYYMMDD IINY-DU-13C -SSIS-YYYYMMDD	None
			899725.64	636601.40					
			899802.30	636786.05					
			899617.66	636862.64					
DU-17	ISM	3	899548.69	637080.83	0–6 in. bgs			IINY-DU-17A-SSIS-YYYYMMDD IINY-DU-17B-SSIS-YYYYMMDD IINY-DU-17C -SSIS-YYYYMMDD	None
			899712.05	637196.09					
			899596.78	637359.38					
			899433.46	637244.19					
DU-18	ISM	3	899433.16	637244.42	0–6 in. bgs			IINY-DU-18A-SSIS-YYYYMMDD IINY-DU-18B-SSIS-YYYYMMDD IINY-DU-18C -SSIS-YYYYMMDD	MS/MSD (extra volume collected) for TOC and CEC from 1 of 3 samples only. MS/MSD for grain size and ORP samples are NA.
			899596.52	637359.69					
			899481.26	637522.97					
			899317.93	637407.79					
DU-19	ISM	3	899333.22	637038.81	0–6 in. bgs			IINY-DU-19A-SSIS-YYYYMMDD IINY-DU-19B-SSIS-YYYYMMDD IINY-DU-19C -SSIS-YYYYMMDD	None
			899496.58	637154.07					
			899381.31	637317.35					
			899217.99	637202.17					

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Iona Island Surface Water Samples									
AOC #16 – Area D (former Dumping Area)	Discrete	20	899537.62	637197.10	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-DUMP-SW-01-YYYYMMDD	Duplicate
			899441.94	637141.79				IINY-DUMP-SW-02-YYYYMMDD	MS/MSD
			899553.24	637254.76				IINY-DUMP-SW-03-YYYYMMDD	None
			899511.81	637224.48				IINY-DUMP-SW-04-YYYYMMDD	None
			899464.15	637196.27				IINY-DUMP-SW-05-YYYYMMDD	None
			899416.05	637169.08				IINY-DUMP-SW-06-YYYYMMDD	None
			899382.58	637144.52				IINY-DUMP-SW-07-YYYYMMDD	None
			899532.76	637290.08				IINY-DUMP-SW-08-YYYYMMDD	None
			899493.66	637258.49				IINY-DUMP-SW-09-YYYYMMDD	None
			899442.31	637226.90				IINY-DUMP-SW-10-YYYYMMDD	None
			899386.05	637200.21				IINY-DUMP-SW-11-YYYYMMDD	None
			899332.24	637178.43				IINY-DUMP-SW-12-YYYYMMDD	None
			899505.81	637323.06				IINY-DUMP-SW-13-YYYYMMDD	None
			899470.48	637294.65				IINY-DUMP-SW-14-YYYYMMDD	None
			899416.43	637268.02				IINY-DUMP-SW-15-YYYYMMDD	None
			899361.09	637235.10				IINY-DUMP-SW-16-YYYYMMDD	None
			899300.29	637210.79				IINY-DUMP-SW-17-YYYYMMDD	None
			899242.55	637199.87				IINY-DUMP-SW-18-YYYYMMDD	None
			899477.89	637354.50				IINY-DUMP-SW-19-YYYYMMDD	None
			899490.49	637170.55				IINY-DUMP-SW-20-YYYYMMDD	None
Hudson River shoreline along eastern boundary of AOC #16 – Area D	Discrete	20	899630.32	637143.37	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-IHRS-SW-01-YYYYMMDD	None
			899627.08	637161.16				IINY-IHRS-SW-02-YYYYMMDD	None
			899623.84	637178.96				IINY-IHRS-SW-03-YYYYMMDD	None
			899620.61	637196.76				IINY-IHRS-SW-04-YYYYMMDD	Duplicate
			899616.21	637214.30				IINY-IHRS-SW-05-YYYYMMDD	MS/MSD
			899611.51	637231.77				IINY-IHRS-SW-06-YYYYMMDD	None
			899606.81	637249.24				IINY-IHRS-SW-07-YYYYMMDD	None
			899602.10	637266.71				IINY-IHRS-SW-08-YYYYMMDD	None
			899594.96	637283.31				IINY-IHRS-SW-09-YYYYMMDD	None
			899587.61	637299.84				IINY-IHRS-SW-10-YYYYMMDD	None
			899579.99	637316.22				IINY-IHRS-SW-11-YYYYMMDD	None
			899569.74	637331.13				IINY-IHRS-SW-12-YYYYMMDD	None
			899559.49	637346.04				IINY-IHRS-SW-13-YYYYMMDD	None
			899549.31	637360.99				IINY-IHRS-SW-14-YYYYMMDD	None
			899539.27	637376.04				IINY-IHRS-SW-15-YYYYMMDD	None
			899529.24	637391.10				IINY-IHRS-SW-16-YYYYMMDD	None
			899519.60	637406.40				IINY-IHRS-SW-17-YYYYMMDD	None
			899510.12	637421.81				IINY-IHRS-SW-18-YYYYMMDD	None
			899499.36	637436.27				IINY-IHRS-SW-19-YYYYMMDD	None
			899487.44	637449.89				IINY-IHRS-SW-20-YYYYMMDD	None

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Iona Marsh shoreline at outlet of riprap filled drainage swale	Discrete	10	898898.81	898898.81	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved (field filtered) mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-RRDS-SW-01-YYYYMMDD	None
			898890.64	898890.64				IINY-RRDS-SW-02-YYYYMMDD	None
			898882.47	898882.47				IINY-RRDS-SW-03-YYYYMMDD	None
			898874.30	898874.30				IINY-RRDS-SW-04-YYYYMMDD	None
			898866.12	898866.12				IINY-RRDS-SW-05-YYYYMMDD	None
			898857.95	898857.95				IINY-RRDS-SW-06-YYYYMMDD	None
			898848.98	898848.98				IINY-RRDS-SW-07-YYYYMMDD	None
			898839.96	898839.96				IINY-RRDS-SW-08-YYYYMMDD	None
			898830.93	898830.93				IINY-RRDS-SW-09-YYYYMMDD	Duplicate
			898821.90	898821.90				IINY-RRDS-SW-10-YYYYMMDD	MS/MSD
Iona Marsh shoreline at outlet of tidal wetland swale	Discrete	10	899261.33	636592.91	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved (field filtered) mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-TWS-SW-01-YYYYMMDD	None
			899249.19	636597.45				IINY-TWS-SW-02-YYYYMMDD	None
			899237.05	636601.99				IINY-TWS-SW-03-YYYYMMDD	None
			899224.91	636606.52				IINY-TWS-SW-04-YYYYMMDD	None
			899212.77	636611.06				IINY-TWS-SW-05-YYYYMMDD	None
			899200.63	636615.60				IINY-TWS-SW-06-YYYYMMDD	None
			899188.49	636620.14				IINY-TWS-SW-07-YYYYMMDD	None
			899176.35	636624.67				IINY-TWS-SW-08-YYYYMMDD	None
			899164.21	636629.21				IINY-TWS-SW-09-YYYYMMDD	None
			899152.08	636633.75				IINY-TWS-SW-10-YYYYMMDD	None
Iona Island ISM Sediment Samples									
IHRS-DU-01 (sized at ½-acre)	ISM	3	899627.39	637142.68	0–6 in. bgs	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-057	Metals by EPA Methods 6010C/6020A Mercury by EPA Method 7471B TOC by Walkley Black Grain size by ASTM D422 pH by field screening ORP by field screening (both field and standard hydrogen electrode corrected values)	IINY-IHRS-DU-01A-SEDIS-YYYYMMDD IINY-IHRS-DU-01B-SEDIS-YYYYMMDD IINY-IHRS-DU-01C-SEDIS-YYYYMMDD	MS/MSD (extra volume collected) for metals, mercury, and TOC samples only. MS/MSD for grain size, pH, and ORP samples are NA.
			899581.03	637181.79					
			899505.71	637533.78					
			899473.84	637545.37					
RRDS-DU-01 (sized at ½-acre)	ISM	3	899010.31	637216.55	0–6 in. bgs			IINY-RRDS-DU-01A-SEDIS-YYYYMMDD IINY-RRDS-DU-01B-SEDIS-YYYYMMDD IINY-RRDS-DU-01C-SEDIS-YYYYMMDD	None
			898949.47	637165.85					
			898724.94	637355.61					
			898764.05	637404.86					
TWS-DU-01 (sized at ½-acre)	ISM	3	899304.36	636542.98	0–6 in. bgs			IINY-TWS-DU-01A-SEDIS-YYYYMMDD IINY-TWS-DU-01B-SEDIS-YYYYMMDD IINY-TWS-DU-01C-SEDIS-YYYYMMDD	None
			899146.47	636474.89					
			899131.98	636608.16					
			899291.32	636671.90					
Iona Island Discrete Sediment AVS/SEM Samples									
IHRS-SED-01		1	899612.05	637216.83	0–6 in. bgs	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-021	AVS/SEM by SM 4500-S2D and EPA Method 6010C	IINY-IHRS-SED-01-YYYYMMDD	None
IHRS-SED-02		1	899580.69	637332.66	0–6 in. bgs			IINY-IHRS-SED-02-YYYYMMDD	None
IHRS-SED-03		1	899530.09	637405.88	0–6 in. bgs			IINY-IHRS-SED-03-YYYYMMDD	None
RRDS-SED-01		1	898948.72	637239.73	0–6 in. bgs			IINY-RRDS-SED-01-YYYYMMDD	Duplicate for AVS/SEM
RRDS-SED-02		1	898839.55	637299.83	0–6 in. bgs			IINY-RRDS-SED-02-YYYYMMDD	None
RRDS-SED-03		1	898759.28	637384.53	0–6 in. bgs			IINY-RRDS-SED-03-YYYYMMDD	None
TWS-SED-01		1	899265.39	636623.00	0–6 in. bgs			IINY-TWS-SED-01-YYYYMMDD	MS/MSD for AVS/SEM
TWS-SED-02		1	899217.33	636572.22	0–6 in. bgs			IINY-TWS-SED-02-YYYYMMDD	None
TWS-SED-03		1	899145.23	636600.05	0–6 in. bgs			IINY-TWS-SED-03-YYYYMMDD	None

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Background Surface Water Samples Con Hook									
Con Hook Hudson River Shoreline	Discrete	20	918638.35	639467.94	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved (field filtered) mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-CHHRS-SW-01-YYYYMMDD	Duplicate
			918628.39	639538.89				IINY-CHHRS-SW-02-YYYYMMDD	MS/MSD
			918583.11	639600.96				IINY-CHHRS-SW-03-YYYYMMDD	None
			918511.59	639631.92				IINY-CHHRS-SW-04-YYYYMMDD	None
			918440.04	639662.80				IINY-CHHRS-SW-05-YYYYMMDD	None
			918368.40	639693.50				IINY-CHHRS-SW-06-YYYYMMDD	None
			918296.50	639723.54				IINY-CHHRS-SW-07-YYYYMMDD	None
			918230.44	639691.91				IINY-CHHRS-SW-08-YYYYMMDD	None
			918162.97	639678.65				IINY-CHHRS-SW-09-YYYYMMDD	None
			918137.31	639739.33				IINY-CHHRS-SW-10-YYYYMMDD	None
			918065.68	639762.80				IINY-CHHRS-SW-11-YYYYMMDD	None
			917988.11	639761.14				IINY-CHHRS-SW-12-YYYYMMDD	None
			917912.03	639745.58				IINY-CHHRS-SW-13-YYYYMMDD	None
			917838.72	639733.25				IINY-CHHRS-SW-14-YYYYMMDD	None
			917783.79	639682.25				IINY-CHHRS-SW-15-YYYYMMDD	None
			917728.77	639634.17				IINY-CHHRS-SW-16-YYYYMMDD	None
			917662.91	639592.79				IINY-CHHRS-SW-17-YYYYMMDD	None
			917588.66	639569.10				IINY-CHHRS-SW-18-YYYYMMDD	None
			917522.56	639559.41				IINY-CHHRS-SW-19-YYYYMMDD	None
			917506.05	639495.13				IINY-CHHRS-SW-20-YYYYMMDD	None
Con Hook Marsh Shoreline	Discrete	20	918430.97	639418.26	NA	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-007 EA-SOP-043	Dissolved (field filtered) TAL metals by EPA Methods 6010C/6020A Dissolved (field filtered) mercury by EPA Method 7470A Hardness by SM2340B Water quality parameters (temperature, pH, specific conductivity, DO, ORP, turbidity, TSS, and salinity) by field screening	IINY-CHMS-SW-01-YYYYMMDD	None
			918392.56	639415.18				IINY-CHMS-SW-02-YYYYMMDD	None
			918364.14	639397.31				IINY-CHMS-SW-03-YYYYMMDD	None
			918329.98	639390.80				IINY-CHMS-SW-04-YYYYMMDD	None
			918291.46	639391.49				IINY-CHMS-SW-05-YYYYMMDD	None
			918252.93	639392.18				IINY-CHMS-SW-06-YYYYMMDD	None
			918214.41	639392.20				IINY-CHMS-SW-07-YYYYMMDD	None
			918175.89	639392.94				IINY-CHMS-SW-08-YYYYMMDD	None
			918137.37	639394.08				IINY-CHMS-SW-09-YYYYMMDD	Duplicate
			918099.04	639397.86				IINY-CHMS-SW-10-YYYYMMDD	MS/MSD
			918060.71	639401.79				IINY-CHMS-SW-11-YYYYMMDD	None
			918022.46	639397.36				IINY-CHMS-SW-12-YYYYMMDD	None
			917984.22	639392.68				IINY-CHMS-SW-13-YYYYMMDD	None
			917947.80	639398.56				IINY-CHMS-SW-14-YYYYMMDD	None
			917913.34	639415.79				IINY-CHMS-SW-15-YYYYMMDD	None
			917880.29	639435.36				IINY-CHMS-SW-16-YYYYMMDD	None
			917848.93	639457.75				IINY-CHMS-SW-17-YYYYMMDD	None
			917813.25	639470.77				IINY-CHMS-SW-18-YYYYMMDD	None
			917775.71	639472.00				IINY-CHMS-SW-19-YYYYMMDD	None
			917737.99	639464.15				IINY-CHMS-SW-20-YYYYMMDD	None

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Background ISM Sediment Samples Con Hook Hudson River Shoreline									
CHHRS-SU-01 (sized at 0.15 acre)	ISM	1	918605.36	639423.29	0–6 in. bgs	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-057	Metals by EPA Methods 6010C/6020A Mercury by EPA Method 7471B TOC by Walkley Black Grain size by ASTM D422 pH by field screening ORP by field screening (both field and standard hydrogen electrode corrected values)	IINY-CHHRS-SU-01-SEDIS-YYYYMMDD	MS/MSD (extra volume collected) for metals, mercury, and TOC samples only. MS/MSD for grain size, pH, and ORP samples are NA.
			918645.76	639420.52					
			918656.83	639582.14					
			918616.42	639584.91					
CHHRS-SU-02 (sized at 0.15 acre)	ISM	1	918582.07	639648.98	0–6 in. bgs			IINY-CHHRS-SU-02-SEDIS-YYYYMMDD	None
			918425.61	639690.97					
			918415.11	639651.85					
			918571.57	639609.87					
CHHRS-SU-03 (sized at 0.15acre)	ISM	1	918398.50	639680.66	0–6 in. bgs			IINY-CHHSR-SU-03-SEDIS-YYYYMMDD	None
			918399.98	639721.13					
			918238.09	639727.06					
			918236.61	639686.59					
CHHRS-SU-04 (sized at 0.15acre)	ISM	1	918187.33	639629.17	0–6 in. bgs			IINY-CHHSR-SU-04-SEDIS-YYYYMMDD	None
			918204.56	639790.25					
			918164.29	639794.56					
			918147.06	639633.48					
CHHRS-SU-05 (sized at 0.15acre)	ISM	1	918132.39	639737.84	0–6 in. bgs			IINY-CHHSR-SU-05-SEDIS-YYYYMMDD	None
			918133.58	639778.32					
			917971.65	639783.08					
			917970.46	639742.60					
CHHRS-SU-06 (sized at 0.15acre)	ISM	1	917950.23	639733.45	0–6 in. bgs			IINY-CHHSR-SU-06-SEDIS-YYYYMMDD	None
			917936.46	639771.54					
			917784.11	639716.47					
			917797.87	639678.38					
CHHRS-SU-07 (sized at 0.15acre)	ISM	1	917778.78	639658.74	0–6 in. bgs			IINY-CHHSR-SU-07-SEDIS-YYYYMMDD	None
			917756.84	639692.78					
			917620.68	639605.00					
			917642.63	639570.96					
CHHRS-SU-08 (sized at 0.15acre)	ISM	1	917606.79	639582.73	0–6 in. bgs			IINY-CHHSR-SU-08-SEDIS-YYYYMMDD	None
			917580.67	639613.68					
			917456.86	639509.21					
			917482.98	639478.26					

Sampling Location	Sample Type	Number of Samples	Northing[1]	Easting[1]	Sample Depth	Sampling Standard Operating Procedure[2]	Analyses	Sample Identification Code[3]	Quality Assurance/Quality Control Samples[3]
Background ISM Sediment Samples Con Hook Marsh Shoreline									
CHMS-SU-01 (sized at 0.15acre)	ISM	1	918415.14	639410.20	0–6 in. bgs	EA-SOP-001 EA-SOP-002 EA-SOP-004 EA-SOP-005 EA-SOP-057	Metals by EPA Methods 6010C/6020A Mercury by EPA Method 7471B TOC by Walkley Black Grain size by ASTM D422 pH by field screening ORP by field screening (both field and standard hydrogen electrode corrected values)	IINY-CHMS-SU-1-SEDIS-YYYYMMDD	None
			918334.79	639399.97					
			918345.03	639319.61					
			918425.38	639329.85					
CHMS-SU-02 (sized at 0.15acre)	ISM	1	918396.64	639297.38	0–6 in. bgs			IINY-CHMS-SU-02-SEDIS-YYYYMMDD	None
			918315.65	639296.28					
			918316.74	639215.29					
			918397.74	639216.39					
CHMS-SU-03 (sized at 0.15acre)	ISM	1	918285.49	639383.86	0–6 in. bgs			IINY-CHMS-SU-03-SEDIS-YYYYMMDD	None
			918204.73	639390.07					
			918198.52	639309.31					
			918279.28	639303.09					
CHMS-SU-04 (sized at 0.15acre)	ISM	1	918154.58	639195.71	0–6 in. bgs			IINY-CHMS-SU-04-SEDIS-YYYYMMDD	None
			918154.46	639276.71					
			918073.46	639276.60					
			918073.58	639195.60					
CHMS-SU-05 (sized at 0.15acre)	ISM	1	918138.54	639385.56	0–6 in. bgs			IINY-CHMS-SU-05-SEDIS-YYYYMMDD	None
			918058.65	639398.95					
			918045.26	639319.07					
			918125.14	639305.67					
CHMS-SU-06 (sized at 0.15acre)	ISM	1	917967.24	639326.68	0–6 in. bgs			IINY-CHMS-SU-06-SEDIS-YYYYMMDD	None
			917991.56	639403.95					
			917914.30	639428.27					
			917889.97	639351.01					
CHMS-SU-07 (sized at 0.15acre)	ISM	1	917908.88	639302.05	0–6 in. bgs			IINY-CHMS-SU-07-SEDIS-YYYYMMDD	None
			917833.04	639330.49					
			917804.60	639254.65					
			917880.44	639226.21					
CHMS-SU-08 (sized at 0.15acre)	ISM	1	917866.16	639441.18	0–6 in. bgs			IINY-CHMS-SU-08-SEDIS-YYYYMMDD	None
			917790.67	639470.54					
			917761.31	639395.05					
			917836.81	639365.69					

1) Northing and Easting coordinates are in New York State Plane Coordinate System, Eastern Zone, NAD 83 (CORS 96) Datum, Feet.
2) Field SOPs that will be used in support of field sampling activities are provided in Appendix H of the UFP-QAPP (EA 2020a) and Appendix D of this UFP-QAPP Addendum.
3) Sample Identification Code for parent samples and duplicate samples is presented in Worksheet #26 and #27.

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
bgs = Below ground surface
CEC = Cation-exchange capacity
DO = Dissolved oxygen
EA = EA Engineering, P.C. and its affiliate EA Science and Technology
EPA = U.S. Environmental Protection Agency
in. = Inch(es)
ISM = Incremental sampling methodology
MS =Matrix spike
MSD = Matrix spike duplicate
NA = Not applicable
ORP = Oxidation-reduction potential
SEM = Simultaneously extracted metals
SIM = Selected ion monitoring
SM = Standard method
SOP = Standard Operating Procedure
TAL = Target Analyte List
TOC = Total organic carbon
TSS = Total suspended solids

UFP-QAPP ADDENDUM WORKSHEET #19 AND #30 – SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

Laboratory: Eurofins TestAmerica Canton, Eurofins TestAmerica Denver, Eurofins Lancaster Laboratories, Inc., Eurofins TestAmerica Corpus Christi, and Katahdin Analytical Services, LLC
Accreditations/Certifications: Provided in Appendix C of this UFP-QAPP Addendum
Sample Delivery Method: Overnight shipping
Data Package Turnaround: Standard Turnaround Time: 21 days for analysis and 21 days for data validation

Matrix	Analytical Group	Analytical/Preparation Method Standard Operating Procedures Reference[1]	Containers (Number, size, and type)	Sample Size[2]	Preservation Requirements (Chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)	Analytical Laboratory
Water	6010C Metals and Hardness	DV-MT-0021 / DV-IP-0010	1, 250 mL, HDPE	100 mL	HNO_3, pH < 2	180 days	Eurofins TestAmerica Denver
	6020A Metals and Hardness	DV-MT-0018 / DV-IP-0014	1, 250 mL, HDPE	100 mL	HNO_3, pH < 2	180 days	Eurofins TestAmerica Denver
	7470A Mercury	DV-MT-0017	1, 250 mL, HDPE	40 mL	HNO_3, pH < 2	28 days	Eurofins TestAmerica Denver
ISM sediment	6010C Metals	DV-MT-0021 / DV-IP-0015	1 kg for multi-incremental sampling, glass or HDPE jar or double-bagged Ziploc bag or bucket	20 g	Cool ≤ 6 °C	180 days	Eurofins TestAmerica Canton for ISM processing Eurofins TestAmerica Denver for analysis
	6020A Metals	DV-MT-0018 / DV-IP-0015		20 g		180 days	Eurofins TestAmerica Canton for ISM processing Eurofins TestAmerica Denver for analysis
	7471B Mercury	DV-MT-0016		5 g		28 days	Eurofins TestAmerica Canton for ISM processing Eurofins TestAmerica Denver for analysis
	Walkley-Black TOC	CA-777		0.5 g		14 days to analysis	Katahdin Analytical Services, LLC
	ASTM D422 Grain Size	CA-551		200 g		None	Katahdin Analytical Services, LLC
Discrete sediment	AVS/SEM (SM 4500-S2D, 6010C)	WI11931; WI11483	4-oz or 8-oz glass jar	100 g	Cool to ≤ 6 ° C	7 days	Eurofins Lancaster Laboratories, Inc.
ISM soil	Walkley-Black TOC	CA-777	1 kg for multi-incremental sampling, glass or HDPE jar or double-bagged Ziploc bag or bucket	10 g	Cool to ≤ 6 ° C	28 days	Katahdin Analytical Services, LLC
	ASTM D422 Grain Size	CA-551		200 g		None	Katahdin Analytical Services, LLC
	SW-846 Method 9081 CEC	CC-ATM-M020		20 g		6 months	Eurofins TestAmerica Corpus Christi

1) Refer to the Analytical SOP References table (Worksheet #23).
2) For discrete samples, the minimum sample size to be provided to the laboratory is indicated, and is based on analysis allowing for sufficient sample for reanalysis. For ISM samples, the subsample volume used by the laboratory is indicated.
3) Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
°C = Degrees Celsius
g = Gram
HDPE = High density polyethylene
ISM = Incremental sampling methodology
kg = Kilogram(s)
HNO_3 = Nitric acid
mL = Milliliter(s)
oz = Ounce
SIM = Selected ion monitoring
SEM = Simultaneously extracted metals
SM = Standard method
TOC = Total organic carbon
ZnAc = Zinc acetate

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UFP-QAPP ADDENDUM WORKSHEET #20 – FIELD QUALITY CONTROL

Table 20-1 ISM Surface Soil Samples

Matrix	Analytical Group	Number of ½-acre DUs	Number of Samples per DU[1]	Maximum Number of MS/MSD[2]	Maximum Total Number of Samples
Surface Soil (0–6 in. bgs)	TOC by Walkley-Black	8	3	2	24
	Grain size by ASTM D422[3]	8	3	0	24
	CEC by EPA SW-846 Method 9081	8	3	2	24
	ORP by field screening	8	3	0	24

1) Each sediment DU or SU will be sampled in triplicate (3 samples per DU or SU).
2) MS/MSD do not count as additional samples. MS/MSD will include extra volume collected at a rate of 1 per 20 samples.

NOTES:
ASTM = American Society for Testing and Materials
bgs = Below ground surface
CEC = Cation-exchange capacity
EPA = U.S. Environmental Protection Agency
in. = Inch(es)
MS = Matrix spike
MSD = Matrix spike duplicate
ORP = Oxidation-reduction potential
TOC = Total organic carbon

Table 20-2 Discrete Surface Water Samples

Matrix	Analytical Group	Maximum Number of Native Samples	Maximum Number of Field Duplicate[1]	Maximum Number of MS/MSD Pairs[2]	Maximum Total Number of Samples
Iona Island surface water	Dissolved TAL Metals by EPA Methods 6010C/6020A	60	3	3	63
	Dissolved Mercury by EPA Method 7470A	60	3	3	63
	Hardness by EPA Method SM 2340B	60	3	3	63
	Field screening for pH, temperature, specific conductivity, DO, ORP, turbidity, TSS, and salinity	60	0	0	60
Con Hook background surface water	Dissolved TAL Metals by EPA Methods 6010C/6020A	40	2	2	42
	Dissolved Mercury by EPA Method 7470A	40	2	2	42
	Hardness by EPA Method SM 2340B	40	2	2	42
	Field screening for pH, temperature, specific conductivity, DO, ORP, turbidity, TSS, and salinity	40	0	0	40

1) Field duplicates will be collected at a rate of 1 per 20 samples.
2) MS/MSD do not count as additional samples. MS/MSD will include extra volume collected at a rate of 1 per 20 samples.
NOTES:
DO = Dissolved oxygen
EPA = U.S. Environmental Protection Agency
MS = Matrix spike
MSD = Matrix spike duplicate
ORP = Oxidation-reduction potential
SM = Standard method
TAL = Target Analyte List
TSS = Total suspended solids

Table 20-3 ISM Sediment Samples

Matrix	Analytical Group	Number of ½-acre DUs	Number of Samples per DU[1]	Maximum Number of MS/MSD[2]	Maximum Total Number of Samples
Iona Island sediment (0–6 in. bgs)	Metals by EPA Methods 6010C/6020A	3	3	1	9
	Mercury by EPA Method 7471B	3	3	1	9
	TOC by Walkley-Black	3	3	1	9
	Grain size by ASTM D422[3]	3	3	0	9
	pH by field screening	3	3	0	9
	ORP by field screening	3	3	0	9
Matrix	**Analytical Group**	**Number of 0.15-acre SUs**	**Number of Samples per SU[1]**	**Maximum Number of MS/MSD[2]**	**Maximum Total Number of Samples**
Con Hook background sediment (0–6 in. bgs)	Metals by EPA Methods 6010C/6020A	16	1	1	16
	Mercury by EPA Method 7471B	16	1	1	16
	TOC by Walkley-Black	16	1	1	16
	Grain size by ASTM D422[3]	16	1	0	16
	pH by field screening	16	1	0	16
	ORP by field screening	16	1	0	16

1) Each sediment DU or SU will be sampled in triplicate (3 samples per DU or SU).
2) MS/MSD do not count as additional samples. MS/MSD will include extra volume collected at a rate of 1 per 20 samples.

NOTES:
bgs = Below ground surface
DU = Decision unit
EPA = U.S. Environmental Protection Agency
in. = Inch(es)
MS = Matrix spike
MSD = Matrix spike duplicate
SU = Sampling unit
TAL = Target Analyte List

Table 20-4 Discrete Sediment AVS/SEM Samples

Matrix	Analytical Group	Maximum Number of Native Samples	Maximum Number of Field Duplicates[1]	Maximum Number of MS/MSD Pairs[2]	Maximum Total Number of Samples
Iona Island sediment (0-6 in. bgs)	AVS/SEM by SM 4500-S2D and EPA Method 6010C	9	1	1	10

1) Field duplicates will be collected at a rate of 1 per 20 samples
2) MS/MSD do not count as additional samples. MS/MSD will include extra volume collected at a rate of 1 per 20 samples.
3) Duplicates and MS/MSD are not applicable for grain size.

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
bgs = Below ground surface
EPA = U.S. Environmental Protection Agency
in. = Inch(es)
MS = Matrix spike
MSD = Matrix spike duplicate
ORP = Oxidation-reduction potential
SEM = Simultaneously extracted metals
SM = Standard method
TOC = Total organic carbon

UFP-QAPP ADDENDUM WORKSHEET #21 – FIELD STANDARD OPERATING PROCEDURES

SOP Reference Number	Responsible Organization	Title, Revision Date and/or Number	Equipment Type or Instrument	Comments
EA-SOP-1	EA	SOP for Sample Labels, November 2018	Sample Labels	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-2	EA	SOP for Chain-of-Custody Form, November 2018	Laboratory supplied chain-of-custody	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-4	EA	SOP for Sampling Packing and Shipping, September 2018	Coolers and shipping materials (bags, tape)	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-5	EA	SOP for Field Decontamination, September 2018	Potable water and cleaning agents	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-7	EA	SOP for Surface Water Sampling, March 2020	Dip sampler	Presented in Appendix D of this UFP-QAPP Addendum
EA-SOP-15	EA	SOP for Document Control System, December 2014	None	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-16	EA	SOP for Surface Water, Groundwater, and Soil/Sediment Field Logbooks	Field book	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-21	EA	SOP for Sediment Sampling, March 2019	Scoops/spoons, logbooks	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-25	EA	SOP for Soil Sampling, December 2014	Scoops/spoons, logbooks	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-43	EA	SOP for Multi-Probe Water Quality Monitoring Instruments, June 2020	Multi-probe instrument	Presented in Appendix D of this UFP-QAPP Addendum[1]
EA-SOP-57	EA	SOP for Incremental Sampling Methodology, March 2020	Incremental sampling tool	Presented in Appendix D of this UFP-QAPP Addendum
EA-SOP-59	EA	SOP for Field Logbook, Revision 1, December 2014	Field logbook	Presented in Appendix H of the UFP-QAPP (EA 2020a)
EA-SOP-64	EA	SOP for Sediment Boring Logs, March 2020	Field logbook	Presented in Appendix D of this UFP-QAPP Addendum

1) Field instruments will be calibrated daily, prior to use. Instruments will be calibrated for parameter(s) to be measured, using standards that bracket the expected concentrations. If the calibration procedure for the method does not allow for the number of standards needed to bracket expected concentrations, the additional standards will be incorporated into a calibration check performed at the start of the day immediately after calibration. A calibration check will also be conducted at the end of each sampling day and when anomalous readings are obtained. Samples analyzed prior to a failing calibration check should be resampled or, if that is not possible, the data should be annotated to indicate that the results obtained may not meet data quality objectives.

NOTES:
EA = EA Engineering, P.C. and its affiliate EA Science and Technology
SOP = Standard Operating Procedure
QAPP = Quality Assurance Project Plan
UFP = Uniform Federal Policy

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UFP-QAPP ADDENDUM WORKSHEET #23 – ANALYTICAL STANDARD OPERATING PROCEDURES REFERENCES TABLE

Laboratory Standard Operating Procedure No.	Title and/or Method Number[1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[2] (Y/N)	Comments
Eurofins TestAmerica Canton, OH							
NC-OP-044 Rev 4	Soil Processing Method ASTM D6323-12, ITRC Guidance Document, Soil Fractionation Method for Michigan	Preparation	ISM Preparation	NA	Eurofins TestAmerica Canton	N	Presented in Appendix E of this UFP-QAPP Addendum
NC-OP-046 Rev. 3	Subsampling, ASTM D6323-12	Preparation	ISM Preparation	NA	Eurofins TestAmerica Canton	N	Presented in Appendix E of this UFP-QAPP Addendum
WI-NC-0238_030521	Soil Processing – Ball Mill Procedure	Definitive	Metals, Sediment	Rolling ball mill by U.S. Stoneware; steel paint cans and ceramic grinding media	Eurofins-TestAmerica Canton	N	Presented in Appendix E of this UFP-QAPP Addendum; duration of milling will be consistent with Phase I samples and consistent between Phase II samples.
Eurofins TestAmerica Denver, CO							
DV-IP-0010 Rev 13	Acid Digestion of Aqueous Samples for Metals Analysis by ICP	Preparation	Metals, Water	NA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-IP-0014 Rev 12	Acid digestion of Aqueous Samples for Analysis by ICPMS (SW-846 3005A, 3020A, and EPA 200.8)	Preparation	Metals, Water	NA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-IP-0015, Rev 14	Acid Digestion of Solids (EPA 3050B)	Preparation	Metals, Soil	NA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-MT-0016 Rev 14	Mercury in Solids by CVAA (SW-846 7471AB)	Definitive	Metals (Mercury), Soil	CVAA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-MT-0017 Rev 14	Mercury in Solids by CVAA (SW-7470A)	Definitive	Metals (Mercury), Water	CVAA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-MT-0018 Rev 11	ICP MS for Trace Element Analysis by SW-846 Method 6020	Definitive	Metals, Water and Soil	ICP	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-MT-0021 Rev 9	ICP Analysis for Trace Elements by SW-846 Method 6010C-D	Definitive	Metals, Water and Soil	ICP	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-OP-0013 Rev 13	Incremental Sampling Methodology for Soils and Sediments (ASTM D 6323)	Preparation	Organic Preparation, Soil	NA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
DV-OP-0015	Microwave Extraction of Solid Samples by Method [SW-846 3546]	Preparation	Organic Preparation, Soil	NA	Eurofins TestAmerica Denver	N	Presented in Appendix E of this UFP-QAPP Addendum
Eurofins Lancaster Laboratories, Inc., PA							
TWCWI11483	Colorimetric Sulfide in Waters, Sulfide as H2S, Dissolved Sulfide in Waters by 4500S2 D2011, 4500S2 H2011, or EPA 376.2	Definitive	AVS, Water	Spectrophotometer	Eurofins Lancaster Laboratories, Inc.	N	Presented in Appendix E of this UFP-QAPP Addendum
T-WC-WI10422	Acid Volatile Sulfide in Solids	Definitive	AVS, Water	NA	Eurofins Lancaster Laboratories, Inc.	N	Presented in Appendix E of this UFP-QAPP Addendum
TMETWI7920	Dilute/Run and AVS/SEM Sample Handling for Metals	Definitive	AVS, Water	NA	Eurofins Lancaster Laboratories, Inc.	N	Presented in Appendix E of this UFP-QAPP Addendum
TMETWI11931	Metals by ICP for Methods SW846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)	Definitive	AVS, Water	Spectrometer	Eurofins Lancaster Laboratories, Inc.	N	Presented in Appendix E of this UFP-QAPP Addendum

Laboratory Standard Operating Procedure No.	Title and/or Method Number[1]	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[2] (Y/N)	Comments
Eurofins TestAmerica Corpus Christi, TX							
CC-ATM-M020	Cation-Exchange Capacity (CEC) of Soils SW-846 Method 9081 and LDNR Method 29-B	Definitive	Cation-Exchange Capacity, Soil	NA	Eurofins TestAmerica Corpus Christi	N	Presented in Appendix E of this UFP-QAPP Addendum
CC-ATM-M005	Trace Metals Analysis by ICP-AES	Definitive	Cation-Exchange Capacity, Soil	NA	Eurofins TestAmerica Corpus Christi	N	Presented in Appendix E of this UFP-QAPP Addendum
Katahdin Analytical Services, LLC, Scarborough, ME							
CA-551-03	Grain Size Analysis, 06/20, Revision 3	Definitive	Solid/Grain Size	Sieves	Katahdin Analytical Services, LLC	N	Presented in Appendix E of this UFP-QAPP Addendum; analysis by sieve and hydrometer
CA-777-00	Titrimetric Determination of Organic Carbon Using the Walkley-Black Method, 05/13, Revision 0	Definitive	Solid/TOC	TOC Analyzer	Katahdin Analytical Services, LLC	N	Presented in Appendix E of this UFP-QAPP Addendum
SD-902-14	Sample Receipt and Internal Control, 05/20, Revision 14	NA	NA	NA	Katahdin Analytical Services, LLC	N	Presented in Appendix E of this UFP-QAPP Addendum
SD-903-06	Sample Disposal, 09/17, Revision 6	NA	NA	NA	Katahdin Analytical Services, LLC	N	Presented in Appendix E of this UFP-QAPP Addendum

1) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.
2) If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy Quality Assurance Officer.

NOTES:
AES = Atomic emission spectrometry
ASTM = ASTM International
AVS = Acid volatile sulfide
CVAA = Cold vapor atomic absorption
EPA = U.S. Environmental Protection Agency
GC/MS = Gas chromatography mass spectrometry
HPLC = High performance liquid chromatography
ICPMS = Inductively coupled plasma mass spectrometry
ISM = Incremental sampling methodology
ITRC = Interstate Technology Regulatory Council
NA = Not applicable
No. = Number
SIM = Selected ion monitoring
TOC = Total organic carbon

UFP-QAPP ADDENDUM WORKSHEET #24 – ANALYTICAL INSTRUMENT CALIBRATION TABLE

This worksheet presents calibration procedures for analytical instruments, including tuning, initial calibration (ICAL), calibration blank, initial calibration verification (ICV) (second source), CCV, linear dynamic range (ICP and ICP/MS only), and verification of detection and quantification limits. Analytical instrument calibration information from Phase I that are applicable to Phase II are provided in the UFP-QAPP (EA 2020a). Calibration information for additional Phase II analyses is provided in this UFP-QAPP Addendum. The following table presents the location of instrument calibration information each Phase II media and laboratory analysis.

Note: Eurofins-TestAmerica Denver analytical instrument calibration information for TAL metals by EPA Methods 6010C/6020A and mercury by 7471B (soil samples) are presented in the UFP-QAPP (EA 2020a). Analytical instrument calibration information for mercury by 7470A (water samples) are the same as for mercury by 7471B (soil samples) presented in the UFP-QAPP (EA 2020a). Hardness by 2340B is a calculation method. The 6010C metals information presented in Worksheet #24 of the UFP-QAPP (EA 2020a) is applicable to the hardness calculation. Katahdin Analytical Services, LLC analytical instrument calibration information for TOC by Walkley-Black is presented in the UFP-QAPP (EA 2020a). There is no calibration information for grain size.

Analytical Method	Location of Measurement Performance Criteria Table
Surface Water Analyses	
Dissolved TAL Metals –Method 6010C	Table 24-1 of the UFP-QAPP (EA 2020a)
Dissolved TAL Metals –Method 6020A	Table 24-2 of the UFP-QAPP (EA 2020a)
Dissolved Mercury –Method 7470A	Table 24-3 of the UFP-QAPP (EA 2020a)
Hardness – SM2340B	NA
Sediment Analyses	
Metals –Method 6010C	Table 24-1 of the UFP-QAPP (EA 2020a)
Metals –Method 6020A	Table 24-2 of the UFP-QAPP (EA 2020a)
Mercury –Method SW7471	Table 24-3 of the UFP-QAPP (EA 2020a)
Sulfide (for AVS/SEM) by SM 4500-S2D-2011	Table 24-1 of this UFP-QAPP Addendum
Metals – ICP Water (for AVS/SEM) by SW-846 6010C	Table 24-1 of this UFP-QAPP Addendum
Total Organic Carbon – Method Walkley-Black	Table 12-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Table 24-8 of the UFP-QAPP (EA 2020a)
Soil Analyses	
Total Organic Carbon – Method Walkley-Black	Table 24-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Table 24-9 of the UFP-QAPP (EA 2020a)
Cation-Exchange Capacity SW-846 Method 9081	Table 24-2 of this UFP-QAPP Addendum

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
ICP = Inductively coupled plasma
NA = Not applicable
SEM = Simultaneously extracted metals
SM = Standard method
TAL = Target Analyte List

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Table 24-1 Analytical Instrument Calibration Table: Sulfide (for AVS/SEM) by SM 4500-S2D-2011 and Metals – ICP Water (for AVS/SEM) by SW-846 6010C (Eurofins Lancaster Laboratories, Inc.)

Parameter/ Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	Standard Operating Procedure Reference[2]
Sulfide / UV Spectro-photometer	Calibration using at least 5 points ranging from 0.10 to 2.0 mg/l	Every 3 months or when a new reagent is prepared	Correlation coefficient must be ≥0.995	Correct the problem and recalibrate	Analyst/ Section Supervisor	WI11483
	CCV Standard	Beginning of each batch, every 10 samples, and end of batch	±10% D	Correct the problem, recalibrate and reanalyze affected samples		
Metals / ICP-AES	ICAL consists of Blank and 1 point: 0 ppm and 50 ppm for Al, Ca, Fe, Mg, K, Na, S, and Si. 0 ppm and 1 ppm for all other metals	Each new run	Passing ICV and ICB	Recalibrate, perform instrument maintenance if calibration cannot conform to criteria, recalibrate	Analyst/ Section Supervisor	WI11931
	ICV	After each calibration	±10% of true value, RSD must be <5%	Recalibrate and reanalyze		
	ICB	Immediately after the ICV	No analytes > 1/2 LOQ	Positive result: accept sample results >10X the ICB or < LOD. Negative result: accept results >10x ICB. All other samples must be reanalyzed with compliant ICB		
	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD		
	CCV	Immediately after the ICS-AB and every 10 samples	±10% of true value, RSD must be <5%	Reanalyze		
	CCB	Immediately after the CCV and at a frequency of every 10 samples	No analytes > 1/2 LOQ	Positive result: accept sample results >10X the ICB or < LOD. Negative result: accept results >10x ICB. All other samples must be reanalyzed with compliant ICB		
	Interference Check Sample-A	At the beginning of each run immediately following the low-level check	Absolute value of concentration for all non-spiked project analytes <1/2 LOQ	Recalibrate		
	Interference Check Sample-AB	At the beginning of each run immediately following the low-level check	± 20% of the true value for each analyte	Recalibrate		
	Low Level Check	Beginning of each sequence and before the interference check samples	± 20% True Value	Reanalyze the sample		
	Linear Range	Quarterly	±10% of true value	Samples > 90% of the linear range must be reanalyzed as a dilution		

1) This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
AES = Atomic emission spectrometry
% = Percent
CCB = Continuing calibration blank
CCV = Continuing calibration verification
D = Difference
ICAL = Initial calibration
ICB = Initial calibration blank
ICS-AB = Interference check solutions AB
ICP = Inductively coupled plasma
ICV = Initial calibration verification
LOD = Limit of detection
LOQ = Limit of quantitation
mg/L = Milligram(s) per liter
ppm = Part(s) per million
RSD = Relative standard deviation

Table 24-2 Analytical Instrument Calibration Table: Cation Exchange Capacity by SW-846 Method 9081 (Eurofins TestAmerica Corpus Christi)

Parameter/ Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	Standard Operating Procedure Reference[2]
ICP-AES	IDL Study	At initial set-up, and after significant change in instrument type, personnel, test method, or sample matrix.	The calculated IDL is verified if the MDLV/DCS standard is detected, and the result is significantly different than the blank.	NA	Laboratory Manager / Analyst	CC-ATM-M005
	IC per manufacturer's instructions, with a minimum of one standard and a calibration blank	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	The validity of the calibration is determined by the subsequent calibration verifications. If invalid, identify and correct problem, then repeat ICAL.		
	Linear dynamic range or high-level check standard	Every 6 months	Within ±10% of the true value for all target analytes.	Adjust dynamic range downward and repeat.		
	ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be <1/2 LOQ or <1/10 the amount measured in any sample, whichever is greater.	Terminate analysis; Correct the problem; Recalibrate.		
	CCV, same source as IC	After the ICV, after every 10 samples and at the end of the run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.		
	CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be < 1/2 LOQ or <1/10 the amount measured in any sample, whichever is greater.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.		

1) This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.
NOTES:
% = Percent
AES = Atomic emission spectrometry
CCB = Continuing calibration blank
CCV = Continuing calibration verification
DCS = Duplicate control sample
IC = Initial calibration
ICAL = Initial calibration
ICB = Initial calibration blank
ICP = Inductively coupled plasma
IDL = Instrument detection limit
RL = Reporting limit
RSD = Relative standard deviation
V = Verification

UFP-QAPP ADDENDUM WORKSHEET #25 – ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

This worksheet presents equipment maintenance, testing, and inspection information for analytical instruments. Equipment maintenance, testing, and inspection information from Phase I that are applicable to Phase II are provided in the UFP-QAPP (EA 2020a). Calibration information for additional Phase II analyses is provided in this UFP-QAPP Addendum. The following table presents the location of instrument calibration information each Phase II media and laboratory analysis.

Note: Eurofins-TestAmerica Denver analytical instrument and equipment maintenance, testing, and inspection information for TAL metals by EPA Methods 6010C/6020A and mercury by 7471B (soil samples) are presented in the UFP-QAPP (EA 2020a). Analytical instrument and equipment maintenance, testing, and inspection information for mercury by 7470A (water samples) are the same as those for mercury analyzed by method 7471B (soil samples) presented in the UFP-QAPP (EA 2020a). Hardness by 2340B is a calculation method. The 6010C metals information presented in Worksheet #25 of the UFP-QAPP (EA 2020a) is applicable to the hardness calculation.

Katahdin Analytical Services, LLC analytical instrument and equipment maintenance, testing, and inspection information for TOC by Walkley-Black, and grain size by ASTM D422 are presented in the UFP-QAPP (EA 2020a).

Analytical Method	Location of Measurement Performance Criteria Table
Surface Water Analyses	
Dissolved TAL Metals –Method 6010C	Worksheet #25 of the UFP-QAPP (EA 2020a)
Dissolved TAL Metals –Method 6020A	Worksheet #25 of the UFP-QAPP (EA 2020a)
Dissolved Mercury –Method 7470A	Worksheet #25 of the UFP-QAPP (EA 2020a)
Hardness – SM2340B	NA
Sediment Analyses	
TAL Metals –Method 6010C	Worksheet #25 of the UFP-QAPP (EA 2020a)
TAL Metals –Method 6020A	Worksheet #25 of the UFP-QAPP (EA 2020a)
Mercury –Method SW7471	Worksheet #25 of the UFP-QAPP (EA 2020a)
Sulfide (for AVS/SEM) by SM 4500-S2D-2011	Table 25-1 of this UFP-QAPP Addendum
Metals – ICP Water (for AVS/SEM) by SW-846 6010C	Table 25-1 of this UFP-QAPP Addendum
Total Organic Carbon –Method Walkley-Black	Worksheet #25 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Worksheet #25 of the UFP-QAPP (EA 2020a)
Soil Analyses	
Total Organic Carbon –Method Walkley-Black	Worksheet #25 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Worksheet #25 of the UFP-QAPP (EA 2020a)
Cation-Exchange Capacity SW-846 Method 9081	Table 25-2 of this UFP-QAPP Addendum

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
ICP = Inductively coupled plasma
NA = Not applicable
SEM = Simultaneously extracted metals
SM = Standard method
TAL = Target Analyte List

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Table 25-1 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table: Sulfide (for AVS/SEM) by SM 4500-S2D-2011 and Metals – ICP Water (for AVS/SEM) by SW-846 6010C (Eurofins Lancaster Laboratories, Inc.)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Standard Operating Procedure Reference[1]
Analytical balance	Assure the balance is in a vibration-free area, is level, and the interior housing is clean.	Verification with ASTM certified weights	Visual inspection and weight verification	Each day of use	The reading must be ±0.1% or ±0.5mg, whichever is greater.	1) verify cleanliness of weights 2) remove balance from service and place a call to service firm 3) management must evaluate data generated since last acceptable reading to determine any potential impacts to data quality	Analyst/ Section Supervisor	QA-SOP-11880
Analytical balance	Annual calibration and maintenance	Annual calibration and maintenance	Annual calibration and maintenance	Annual	As per vendor's specifications in compliance with ISO certification	As per vendor's specifications in compliance with ISO certification	Professional calibration vendor (ISO 17025 certified)	QA-SOP-11880
UV Spectrophotometer	As needed replacement of components	Calibration checks	Visual inspection of components	As needed maintenance/calibration checks every 10 injections	90-110% for calibration checks	Recalibration	Analyst/ Section Supervisor	WI11483
Thermo 6000 Series ICP-AES	As needed replacement of components	Calibration checks	Visual inspection of components	As needed maintenance/ calibration checks every 10 injections	90-110% for the calibration checks	Recalibration	Analyst/ Section Supervisor	WI11931

1) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.
NOTES:
% = percent
AES = Atomic emission spectrometry
ASTM = ASTM International
ICP = Inductively coupled plasma
ISO = International Organization for Standardization
mg = Milligram(s)
QA = Quality assurance
SOP = Standard Operating Procedure
UV = Ultraviolet

Table 25-2 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table: Cation Exchange Capacity by SW-846 Method 9081 (Eurofins TestAmerica Corpus Christi)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Standard Operating Procedure Reference[1]
ICP	Check all pump tubing	None	Preventative Maintenance	Daily	None	None	Eurofins Xenco Chemist	CC-ATM-M005
ICP	Check/clean torch, glassware, and injector	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	None	Clean torch, glassware, and injector	Eurofins Xenco Chemist	CC-ATM-M005
ICP	Check air filters and instrument exterior	None	Instrument performance	Daily	None	Replace filters when needed	Eurofins Xenco Chemist	CC-ATM-M005
ICP	Clean/Replace lenses	None	Instrument performance	As needed	None	Clean or Replace lenses	Eurofins Xenco Chemist	CC-ATM-M005

1) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.
NOTES:
ICP = Inductively coupled plasma
SOP = Standard Operating Procedure

UFP-QAPP ADDENDUM WORKSHEET #26 AND #27 – SAMPLE HANDLING, CUSTODY, AND DISPOSAL

To ensure sample authenticity and data defensibility, a proper sample handing system will be followed from the time of sample collection to final sample disposal.

Sampling organization: EA Engineering, P.C. and its affiliate EA Science, and Technology.

Laboratory:

- TestAmerica Canton, OH (ISM sediment preparation)
- Eurofins TestAmerica, Denver, CO (surface water and sediment metals and hardness)
- Eurofins-Lancaster, PA (Sediment AVS/SEM analysis)
- Eurofins TestAmerica, Corpus Christi, TX (Soil CEC)
- Katahdin Analytical Services, LLC, Scarborough, ME (Soil and sediment TOC and grain size analyses).

Method of sample delivery (shipper/carrier):

- Overnight Shipping (Federal Express or United Parcel Service).

Number of days from reporting until sample disposal: Per laboratory requirements.

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference[1]
Sample labeling	EA, field personnel	EA-SOP-1
Chain-of-custody form completion	EA, field personnel	EA-SOP-2
Packaging	EA, field personnel	EA-SOP-4
Shipping coordination	EA, field personnel	EA-SOP-4
Sample receipt, inspection, and log-in	Laboratory sample log-in staff and laboratory PM	TBD
Sample custody and storage	Laboratory PM	TBD
Sample disposal	Laboratory Waste Manager	TBD

1) Field SOPs that will be used in support of field sampling activities are included in Appendix H of the UFP-QAPP (EA 2020a).
NOTES:
EA = EA Engineering, P.C. and its affiliate EA Science and Technology
PM = Project Manager
SOP = Standard Operating Procedure
TBD = To be determined

26.1 FIELD SAMPLE CUSTODY PROCEDURES (SAMPLE COLLECTION, PACKAGING, SHIPMENT, AND DELIVERY TO LABORATORY)

Sample Identification—Weatherproof sample labels with sample identification No. will be affixed to each sample container. Each sample will be assigned a unique alpha-numeric code to assist in the tracking of samples and to facilitate retrieval of analytical results. The sampling code will be used on sample labels, sample tracking forms, chain of custody forms, field logs, and any other applicable documentation.

The alpha-numeric code for ISM samples will be as follows:

- Site name-sample location unit identifier-triplicate sample indicator-media identifier-date code

  — Site name = IINY for Iona Island, New York

  — Sample location unit identifier as presented in Worksheet #11 and #18 (DU-## for onsite soil DUs; IHRS-DU-01 for Iona Island Hudson River Shoreline DU; RRDS-DU-01 for Iona Island riprap filled drainage swale DU; TWS-DU-01 for Iona Island tidal wetland swale DU; CHHRS-SU-XX for Con Hook Hudson River shoreline SUs; CHMS-SU-XX for Con Hook marsh shoreline SUs)

  — Triplicate sample indicator for ISM sediment samples = A for initial sample, B for duplicate sample, or C for triplicate sample.

  — Media identifier includes SSIS for surface soil incremental samples and SEDIS for sediment incremental samples

  — Date code = the six-digit date codes correspond to YYYYMMDD. For example, 20220708 corresponds to samples collected on 8 July 2022.

The alpha-numeric code for discrete samples will be as follows:

- Site name-sample location identifier- -media identifier-sample number-date code

  — Site name = IINY for Iona Island, New York

  — Sample location identifier as presented in Worksheet #11 and #18 (DUMP for AOC #16 – Area D [former Dumping Area]; IHRS for Iona Island Hudson River Shoreline ; RRDS for Iona Island riprap filled drainage swale; TWS for Iona Island tidal wetland swale; CHHRS for Con Hook Hudson River shoreline; CHMS for Con Hook marsh shoreline)

  — Media identifier includes SW for surface water and SED for sediment

— Sample number as presented in Worksheet #11 and #18 (01 to 20 for discrete surface water samples and 01 to 03 for discrete sediment samples)

— Date code = the six-digit date codes correspond to YYYYMMDD. For example, 20220708 corresponds to samples collected on 8 July 2022.

For example, surface water samples, ISM sediment samples, and discrete sediment AVS/SEM samples collected along the Iona Hudson River Shoreline on 8 July 2022 would be labeled as follows:

- Surface water samples: IINY-IHRS-SW-01-20220708 to IINY-IHRS-SW-20-20220708
- ISM sediment samples: IINY-IHRS-DU-01A-SEDIS-20220708; IINY-IHRS-DU-01B-SEDIS-20220708; IINY-IHRS-DU-01C- SEDIS-20220708
- Discrete sediment AVS/SEM samples: IINY-IHRS-SED-01-20220708; IINY-IHRS-SED-02-20220708; IINY-IHRS-SED-03-20220708

The sample duplicates for surface water and discrete sediment AVS/SEM samples will follow a naming convention to be blind to the laboratory:

- Site name-Duplicate code-Media Identifier- Date Code- Consecutive No. code

  — Site name = IINY for Iona Island, New York

  — Duplicate code = DUP

  — Media identifier = SW for surface water, SED for sediment locations

  — Date code = the six-digit date codes correspond to YYYYMMDD. For example, 20220708 corresponds to samples collected on 8 July 202w.

  — Consecutive No. code to distinguish samples if multiple duplicates are collected in a given day = 1, 2, 3, etc.

For example, the first surface water and discrete sediment AVS/SEM duplicate samples collected on 8 July 2022 would be labeled as follows:

- IINY-DUP-SW-20220708-01
- IINY- DUP-SED-20220708-01

26.2 SAMPLE TRACKING

Sample tracking will be conducted as presented in Worksheet #26 and #27 of the UFP-QAPP (EA 2020a).

26.3 SAMPLE CUSTODY AND DOCUMENTATION

Sample custody and documentation will be conducted as presented in Worksheet #26 and #27 of the UFP-QAPP (EA 2020a).

26.4 SAMPLE PACKAGING AND SHIPPING

Sample packaging and shipping will be conducted as presented in Worksheet #26 and #27 of the UFP-QAPP (EA 2020a).

UFP-QAPP ADDENDUM WORKSHEET #28 – QUALITY CONTROL SAMPLES TABLE

This worksheet presents analytical quality control and corrective action information for laboratory analyses. The purpose of this worksheet is to ensure that the selected analytical methods are capable of meeting project-specific measurement performance criteria, which are based on DQOs. Analytical quality control and corrective action information from Phase I media and analyses that are applicable to Phase II are provided in the UFP-QAPP (EA 2020a). Analytical quality control and corrective action information for additional Phase II media and analyses are provided in this UFP-QAPP Addendum. The following table presents the location of instrument calibration information each Phase II media and laboratory analysis.

Note: Eurofins-TestAmerica Denver QC and corrective action information for TAL metals in sediment samples by EPA Methods 6010C/6020A and mercury in sediment samples by 7471B are presented in the UFP-QAPP (EA 2020a).

Hardness by 2340B is a calculation method. The QC and corrective action information for 6010C metals presented in the UFP-QAPP (EA 2020a) is applicable to the hardness calculation results.

Katahdin Analytical Services, LLC measurement performance criteria tables for TOC by Walkley-Black are presented in the UFP-QAPP (EA 2020a). There are no measurement performance criteria tables for grain size.

Analytical Method	Location of Quality Control Samples Table
Surface Water Analyses	
Dissolved TAL Metals –Method 6010C	Table 28-1 of this UFP-QAPP Addendum
Dissolved TAL Metals –Method 6020A	Table 28-2 of this UFP-QAPP Addendum
Dissolved Mercury –Method 7470A	Table 28-3 of this UFP-QAPP Addendum
Hardness – SM2340B	NA
Sediment Analyses	
Metals –Method 6010C	Table 28-1 of the UFP-QAPP (EA 2020a)
Metals –Method 6020A	Table 28-2 of the UFP-QAPP (EA 2020a)
Mercury –Method SW7471	Table 28-3 of the UFP-QAPP (EA 2020a)
Sulfide (for AVS/SEM) by SM 4500-S2D-2011	Tables 28-4 and 28-5 of this UFP-QAPP Addendum
Metals – ICP Water (for AVS/SEM) by SW-846 6010C	Table 28-6 of this UFP-QAPP Addendum
Total Organic Carbon –Method Walkley-Black	Table 28-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Table 28-9 of the UFP-QAPP (EA 2020a)
Soil Analyses	
Total Organic Carbon –Method Walkley-Black	Table 28-8 of the UFP-QAPP (EA 2020a)
Grain Size Method ASTM D422	Table 28-9 of the UFP-QAPP (EA 2020a)
Cation-Exchange Capacity SW-846 Method 9081	Tables 28-7 and 28-8 of this UFP-QAPP Addendum

NOTES:
ASTM = ASTM International
AVS = Acid volatile sulfide
ICP = Inductively coupled plasma
NA = Not applicable
SEM = Simultaneously extracted metals
SM = Standard method
TAL = Target Analyte List
UFP = Uniform Federal Policy

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Table 28-1 Quality Control Samples: Target Analyte List Metals Method 6010C (Eurofins TestAmerica Denver)

Matrix: Water
Analytical Group: Metals
Analytical Method/SOP Reference[2]: EPA 6010C DV-MT-0021

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedure Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1/ Preparatory Batch (20 samples)	No Target Compounds> ½ LOQ or greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Common lab contaminants: no analytes detected > LOQ.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x negative blanks or sample results ND.	Analyst/Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>1/2 LOQ
LCS	1/Preparatory Batch (20 samples)	DoD QSM 5.3 recovery limits.	If acceptable, report. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and samples in the associated prep batch for failed analytes, if sufficient sample material is available. Marginal exceedance allowed unless analyte is specified risk driver.	Analyst/Section Supervisor	Accuracy/Bias	DoD QSM 5.3 or Laboratory % Recovery / RPD Control Limits
MS/MSD	1 pair/Preparatory Batch (20 samples)	Recovery: DoD QSM 5.3 limits. RPD: RPD between MS and MSD < 20%	If MS fails, consult project specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error. For Sample/Matrix Duplicate criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.	Analyst/Section Supervisor	Accuracy/Bias/ Precision	DoD QSM 5.3 or Laboratory % Recovery/RPD Control Limits
Dilution test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations >50 x LOQ. Use along with MS/MSD and PDS data to confirm matrix effects	Five-fold dilution must agree within + 10% of the original determination	If dilution test fails analyze post digestion spike.	Analyst Section Supervisor	Accuracy/Bias/ Precision	N/A
Post digestion spike addition	When MS/MSD fails and analyte concentration < 50 x LOQ	Recovery within 80-120% of expected results	For specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Section Supervisor	Accuracy/Bias	N/A
Method of Standard Additions	When dilution test or PDS fails and if required by the project	NA	Document use of MSA in Case Narrative	Analyst/Section Supervisor	Accuracy/Bias	N/A
LOQ Check	Quarterly	Compound specific recovery limits included in Appendix E	Per DoD QSM requirements	Section Supervisor/QA Manager	Accuracy/Sensitivity	Compound Specific Recovery Limits

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:

% = percent	LOQ = Limit of quantitation	NA = Not applicable	QSM = Quality Systems Manual
DoD = Department of Defense	MS = Matrix spike	ND = Non-detect	RPD = Relative percent difference
DQO = Data quality objective	MSA = Measurement systems analysis	PDS = Post digestion spike	UFP = Uniform Federal Policy
LCS = Laboratory control sample	MSD = Matrix spike duplicate	QAPP = Quality Assurance Project Plan	

Table 28-2 Quality Control Samples: Target Analyte List Metals Method 6020A (Eurofins TestAmerica Denver)

Matrix: Water
Analytical Group: Metals
Analytical Method/SOP Reference:[2] EPA 6020A DV-MT-0018

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedures Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1/ Preparatory Batch (20 samples)	The absolute values of analytes must be < ½ LOQ or 1/10 the regulatory limit (whichever is greater). Common lab contaminants: no analytes detected > LOQ. Non-detects associated with positive blank infractions may be reported. Sample results >10x the LOQ associated with negative blanks may be reported.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst/Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>1/2 LOQ
LCS	1/Preparatory Batch (20 samples)	DoD QSM 5.3 recovery limits.	If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and samples in the associated prep batch for failed analytes, if sufficient sample material is available. Marginal exceedances allowed unless analyte is specified risk driver.	Analyst/Section Supervisor	Accuracy/Bias	DoD QSM 5.3 or Laboratory % Recovery/RPD Control Limits
MS/MSD	1 pair/Preparatory Batch (20 samples)	Recovery: DoD QSM 5.3 limits. RPD: RPD between MS and MSD < 20% For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.	If MS fails, consult project specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst/Section Supervisor	Accuracy/Bias/ Precision	DoD QSM 5.3 or Laboratory % Recovery/RPD Control Limits
Dilution test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations >50 x LOQ. Use along with MS/MSD or PDS data to confirm matrix effects.	Five-fold dilution must agree within + 10% of the original determination	If dilution test fails analyze post digestion spike.	Analyst/Section Supervisor	Accuracy/Bias/ Precision	NA
Post digestion spike addition	When MS/MSD fails or analyte concentration of samples < 50 x LOQ.	Recovery within 80-120% of expected results	For specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Section Supervisor	Accuracy/Bias	NA
Method of Standard Additions	When dilution test or post digestion spike fails and if required by the project	NA	Document use of MSA in the Case Narrative	Analyst/Section Supervisor	Accuracy/Bias	NA
LOQ Check	Quarterly	Compound specific recovery limits included in Appendix E	Per DoD QSM requirements	Section Supervisor/QA Manager	Accuracy/Sensitivity	Compound Specific Recovery Limits

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
% = Percent
DoD = Department of Defense
DQO = Data quality objective
LCS = Laboratory control sample
LOQ = Limit of quantitation
MS = Matrix spike
MSA = Measurement systems analysis
MSD = Matrix spike duplicate
NA = Not applicable
ND = Non-detect
PDS = Post digestion spike
QAPP = Quality Assurance Project Plan
QSM = Quality Systems Manual
RPD = Relative percent difference
UFP = Uniform Federal Policy

Table 28-3 Quality Control Samples: Mercury Methods 7470A (Eurofins TestAmerica Denver)

Matrix: Water
Analytical Group: Mercury
Analytical Method/SOP Reference:[2] EPA 7470A, DV-MT-0017

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedure Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1/Preparatory Batch (20 samples)	Absolute value of analyte must be < ½ LOQ or < 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Correct problem then re-prep and analyze method blank and samples processed with the contaminated blank. Non-detects associated with positive blank results may be reported. Sample results > 10x the LOQ associated with negative blanks may be reported. If reanalysis is not possible, apply B-flag to results for the specific analyte(s) in samples processed with the contaminated blank. Must be explained in the case narrative.	Analyst/Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>1/2 RL
LCS	1/Preparatory Batch (20 samples)	DoD QSM 5.3 limits	If the LCS recovery is above the project acceptance limits and there are no detections in the samples, report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR) Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in samples in associated batch.	Analyst/Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
MS/MSD	1/Preparatory Batch (20 samples)	DoD QSM 5.3 Limits RPD: < 20% For Sample/MD: % recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.	If MS fails, consult project specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Analyst/Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
LOQ Check	Quarterly	Compound specific recovery limits included in Appendix E	Per DoD QSM requirements	Section Supervisor/QA Manager	Accuracy/Sensitivity	Compound Specific Recovery Limits

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
% = percent
DoD = Department of Defense
DQO = Data quality objective
LCS = Laboratory control sample
LOQ = Limit of quantitation
MS = Matrix spike
MSD = Matrix spike duplicate
ND = Non-detect
QAPP = Quality Assurance Project Plan
QSM = Quality Systems Manual
RPD = Relative percent difference
RL = Reporting limit
UFP = Uniform Federal Policy

Table 28-4 Quality Control Samples: Sulfide (for AVS/SEM) by SM 4500-S2D-2011 (Eurofins Lancaster Laboratories, Inc.)

Matrix: Sediment AVS/SEM extract
Analytical Group: Sulfide
Analytical Method/SOP Reference:[2] SM 4500-S2D-2011; WI11483

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedure Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method blank	1 per prep batch of up to 20 samples	No analytes detected > 1/2 LOQ or >1/10 the amount measured in any sample.	Reanalyze blank to confirm detections. If detects confirm, reanalyze samples that are not ND or not >10x the blank value.	Analyst/ Section Supervisor	Accuracy/Laboratory Contamination	No analytes detected > 1/2 LOQ or >1/10 the amount measured in any sample.
MS/MSD	1 per 20 samples	57-108% %R, RPD≤20%	Flag outliers	Analyst/ Section Supervisor	Accuracy/Bias/Precision	Results within acceptance limits
LCS	1 per prep batch of up to 20 samples	57-108% %R	Reanalyze LCS and associated samples. Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-analyzed.	Analyst/ Section Supervisor	Accuracy/Bias/Precision	Results within acceptance limits
Duplicate	1 per 20 samples	RPD≤20%	Flag data	Analyst/ Section Supervisor	Precision	Results within acceptance limits

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
LCS = Laboratory control sample
LOQ = Limit of quantitation
MS = Matrix spike
MSD = Matrix spike duplicate
ND = Non-detect
QAPP = Quality Assurance Project Plan
RPD = Relative percent difference
UFP = Uniform Federal Policy

Table 28-5 Quality Control Samples: Water Metals (for AVS/SEM) by SW-846 6010C (Eurofins Lancaster Laboratories, Inc.)

Matrix: Sediment AVS/SEM extract
Analytical Group: Metals
Analytical Method/SOP Reference[2]: SW-846 6010C; WI11931

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedure Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per prep batch of up to 20 samples	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	Analyst/ Section Supervisor	Accuracy/Laboratory Contamination	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Analyze post digestion spike and serial dilution	Analyst/ Section Supervisor	Accuracy/Bias/Precision	Results within acceptance limits
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-digested and reanalyzed.	Analyst/ Section Supervisor	Accuracy/Bias/Precision	Results within acceptance limits
Duplicate	1 per prep batch of up to 20 samples	RPD must be ≤20%	Flag data	Analyst/ Section Supervisor	Precision	Results within acceptance limits
Serial Dilutions	Must be prepared with each background sample, evaluated only when analyte concentrations are >50x the LOQ	The percent difference must be ≤10%	Flag data	Analyst/ Section Supervisor	Precision	Results within acceptance limits
PDS	Prepare with each background sample	± 20% True Value	No specific action needed unless required by the project. PDS is reported in data package	Analyst/ Section Supervisor	Accuracy/Bias	Results within acceptance criteria
Internal Standard	Every sample and QC	Must be 50%-130% of the calibration blank	Reanalyze at a dilution	Analyst/ Section Supervisor	Precision	Results within acceptance limits

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
% = Percent
LCS = Laboratory control sample
LCSD = Laboratory control sample duplicate
LOQ = Limit of quantitation
MS = Matrix spike
MSD = Matrix spike duplicate
ND = Non-detect
PDS = Post digestion spike
QAPP = Quality Assurance Project Plan
QC = Quality control
QSM = Quality systems manual
RL = Reporting limit
RPD = Relative percent difference
SOP = Standard Operating Procedure
UFP = Uniform Federal Policy

Table 28-6 Quality Control Samples: Cation-Exchange Capacity SW-846 Method 9081 (Eurofins TestAmerica Corpus Christi)

Matrix: Soil
Analytical Group: Cation-Exchange Capacity
Analytical Method/SOP Reference[2]: **SW-846 Method 9081 and Method 6010B**

Quality Control Sample	Frequency/Number	Method/Standard Operating Procedure Quality Control Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator
Method blank	One per digestion batch of up to 20 samples	No results >1/2 LOQ or >1/10 the amount measured in any sample.	Rerun once in a new tube. If >RL, re-digest and reanalyze samples. Note exceptions under criteria section.	Lab Manager / Analyst	Accuracy/Bias Contamination
ICSA	At the beginning of the analytical run	The absolute value of ICSA results for the non-interfering elements must be ≤ ½ LOQ.	The IECs must be re-evaluated and corrected if necessary and the affected samples re-analyzed, or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.	Lab Manager / Analyst	Accuracy
LCS	One LCS per each preparation batch of up to 20 samples	87-120 % recovery	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the LCS.	Lab Manager / Analyst	Precisions and Accuracy/Bias
MS/MSD for all analytes	Every 10%, at least one per prep batch	87-115% recovery; MSD: RPD ≤ 20%	Flag the data and document in the case narrative; no flag required if the sample level is > 4x the spike added.	Lab Manager / Analyst	Precision and Accuracy/Bias
Dilution test	One per prep batch	1:5 dilution must agree within ±10% of the original determination in accordance with DoD QSM requirements	For samples > 50x MDL, dilutions must agree within 10%.	Lab Manager / Analyst	Accuracy
Post digestion spike addition	Analyzed if MS/MSD fails, one per prep batch	Recovery for the PDS should be within 80-120% of the expected results.	Flag the data and document in the case narrative.	Lab Manager / Analyst	Accuracy

1) This is a summary of the acceptance criteria; refer to Appendix E of this UFP-QAPP Addendum for specific or more information.
2) SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

NOTES:
% = Percent
µg/L = Microgram(s) per liter
DoD = Department of Defense
ICSA = Interference check analysis
LCS = Laboratory control sample
MS = Matrix spike
MSD = Matrix spike duplicate
MDL = Method detection limit
PDS = Post digestion spike
QAPP = Quality Assurance Project Plan
QSM = Quality systems manual
RL = Reporting limit
UFP = Uniform Federal Policy

Table 28-7 Quality Cation-Exchange Capacity SW-846 Method 9081 Recovery and Precision Limits (Eurofins TestAmerica Corpus Christi)

Analyte	Precision (RPD)	Recovery Limits (LCS/MS/MSD)
Aluminum	20	80-120
Antimony	20	80-120
Arsenic	20	80-120
Barium	20	80-120
Beryllium	20	80-120
Bismuth	20	80-120
Boron	20	80-120
Calcium	20	80-120
Chromium	20	80-120
Cobalt	20	80-120
Copper	20	80-120
Iron	20	80-120
Lead	20	80-120
Manganese	20	80-120
Molybdenum	20	80-120
Nickel	20	80-120
Phosphorus	20	80-120
Potassium	20	80-120
Selenium	20	80-120
Silicon	20	80-120
Silica	20	80-120
Silver	20	80-120
Sodium	20	80-120
Strontium	20	80-120
Thallium	20	80-120
Tin	20	80-120
Titanium	20	80-120
Uranium	20	80-120
Vanadium	20	80-120
Zinc	20	80-120

1) Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory.
NOTES:
LCS = Laboratory control sample
MS = Matrix spike
MSD = Matrix spike duplicate
RPD = Relative percent difference

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UFP-QAPP ADDENDUM WORKSHEET #36 – DATA VALIDATION PROCEDURES

Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods/SOPs, and method performance criteria. The validation to be performed for each data type is summarized in this worksheet. Data validation will be performed by the EA Project Chemist.

Data Validation

Analytical Group/Method	TAL metals by EPA Method 6010C/6020A, mercury by EPA Method 7470A/7471B, hardness by SM2340B, AVS/SEM by SM 4500-S2D and EPA Method 6010C, TOC by Walkley-Black, CEC by EPA SW-846 Method 9081
Data deliverable requirements	Level 4 PDF data package; SEDD format Stage 2A version 5.2
Analytical specifications	Worksheet #28
Measurement performance criteria	Worksheet #12
Percent of data packages to be validated	100%
Percent of raw data reviewed	0%
Percent of results to be recalculated	0%
Validation procedure	FUDSChem eQAPP; DoD-QSM, version 5.3; and EPA National Functional Guidelines
Validation code[1]	S2bVEM
Electronic validation program/version	ADR in FUDSChem

1) Refer to Validation Code and Label Identifier Table.
NOTES:
% = percent
AVS = Acid volatile sulfide
ADR = Automatic Data Review
CEC = Cation-exchange capacity
DoD = Department of Defense
EPA = U.S. Environmental Protection Agency
FUDSChem = Formerly Used Defense Sites Online Chemistry Database
SEDD = Staged electronic data deliverables
SEM = Simultaneously extracted metals
SIM = Selective ion monitoring
SM = Standard method
QSM = Quality Systems Manual
TAL = Target Analyte List

Validation Code and Label Identifier Table

Validation Code	Validation Label	Description	Reference
S1VE	Stage 1 Validation Electronic	Stage 1 Validation - Verification and validation based only on completeness and compliance of sample receipt condition checks.	EPA 540-R-08-005
S1VM	Stage 1 Validation Manual		
S1VEM	Stage 1 Validation Electronic and Manual		
S2aVE	Stage 2a Validation Electronic	Stage 2A Validation - Verification and validation based on completeness and compliance checks of sample receipt conditions and ONLY sample-related QC results.	
S2aVM	Stage 2a Validation Manual		
S2aVEM	Stage 2a Validation Electronic and Manual		
S2bVE	Stage 2b Validation Electronic	Stage 2B Validation - Verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results.	
S2bVM	Stage 2b Validation Manual		
S2bVEM	Stage 2b Validation Electronic and Manual		
S3VE	Stage 3 Validation Electronic	Stage 3 Validation - Verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, AND recalculation checks.	
S3VM	Stage 3 Validation Manual		
S3VEM	Stage 3 Validation Electronic and Manual		
S4VE	Stage 4 Validation Electronic	Stage 4 Validation - Verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs.	
S4VM	Stage 4 Validation Manual		
S4VEM	Stage 4 Validation Electronic and Manual		
NV	Not Validated	NA	

NOTES:
EPA = U.S. Environmental Protection Agency
NA = Not applicable
NV = Not validated
VE = Validation electronic
VEM = Validation electronic and manual

The following data qualifiers will be applied during data validation. Potential impacts on project specific DQOs will be discussed in the data validation report.

- NM – Measurement Performance Criteria contained in Worksheet #12 were not met.
- J – The result is an estimated value. The nature of the bias will be discussed in the data validation report.

- J- – The result is an estimated value with a potential low bias.
- J+ – The result is an estimated value with a potential high bias.
- X – The sample results were affected by serious deficiencies. The analyte may or may not be present in the sample. Exclusion of the data is recommended.
- R – The data are unusable and are rejected.
- U – The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the LOD.
- UJ – The analyte was analyzed for but was not detected above the level of the associated value. The associated value is an estimate and may be inaccurate or imprecise.

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UFP-QAPP Addendum Appendix A

Phase I HTRW Technical Memorandum

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4 September 2020

TECHNICAL MEMORANDUM

TO: Erin Kirby, USACE NAE

FROM: Mike McGuire, EA

CC: Todd Beckwith, USACE NAB
Amanda Kohn, EA

SUBJECT: Iona Island Phase I HTRW Technical Memorandum

The Hazardous Toxic and Radioactive Waste (HTRW) Remedial Investigation (RI) is being conducted to characterize the nature and extent of hazardous substances in environmental media attributable to past Department of Defense (DoD) activities and evaluate potential risk to human health and ecological receptors. On 9-20 March 2020 and 30 March through 1 April 2020, EA Engineering, Science, and Technology, Inc., PBC (EA) conducted the Phase I HTRW project RI field activities at the Iona Island Naval Ammunition Depot Formerly Used Defense Site (FUDS) located in Stony Point, Rockland County, New York along the west bank of the Hudson River (**Figure 1**). Phase I field activities were conducted in accordance with the HTRW RI Uniform Federal Policy (UFP) – Quality Assurance Project Plan (QAPP) (EA 2020a). The purpose of this Phase I HTRW Technical Memorandum is to present the Phase I field activities that were completed in March and April 2020 and present tabulated laboratory analytical results. Further evaluation and reporting of results will be provided in the Phase I Data Report, to be submitted at a later date.

Investigations have been ongoing at Iona Island since 1992. Two previous investigations include the 1996 Data Collection Activities (Greeley-Polhemus 1997) and 2007 Site Inspection (Alion Science and Technology 2008), which reported widespread detections of metals and polycyclic aromatic hydrocarbons (PAHs) in surface soil and/or subsurface soil at the Iona Island Naval Ammunition Depot FUDS. Concentrations of metals and PAHs at several locations were reported above U.S. Environmental Protection Agency (EPA) screening levels as presented in the UFP-QAPP (EA 2020a)[1] (**Figure 2**). Nineteen areas of concern (AOCs) were identified as eligible for further inclusion in the C02NY074402 HTRW project, based on previous documents, aerial photographs, and available information. These FUDS-eligible AOCs encompass the locations/footprints of former buildings/structures where contaminants related to the facilities or from historical processes during the site's commission as an ammunition depot may be present,

[1] EPA Industrial and/or Residential Soil Regional Screening Levels (RSLs) (updated May 2020), EPA Ecological Soil Screening Levels (EcoSSLs) (February 2005 through April 2008), and/or EPA Region 4 Marine/Estuarine Sediment Screening Benchmarks (EPA 2018).

including 16 former buildings, 4 former above-ground storage tanks (ASTs)[2], a former dumping area, 2 former coal storage areas, and 3 former transformer locations:

- AOC #1: Former Building 103 – Paint and Oil Storage
- AOC #2: Former Building 121 – Incinerator
- AOC #3: Former Building 123 – Garage Building
- AOC #4: Former Building 124 to 128 – Sewage Disposal Plant (and Suspected Outfall)
- AOC #5: Former Building 207 – Marine Garage Building
- AOC #6: Former Building 213 – Paint Locker for Building 202
- AOC #7: Former Building 215 – Garage Building
- AOC #8: Former Building 219 – Garage Building
- AOC #9: Former Building 220 – Garage Building
- AOC #10: Former Building 406 – Tin, Electrical, and Annealing Building Shop
- AOC #11: Former Building 407 – Paint Shop and Pipe Shop
- AOC #12: Former Building 410 – Power House
- AOC #13: Area A – One Former Fuel Oil AST (2,500 gallons); located near Former Building 223
- AOC #14: Area B – One Former Fuel Oil AST (size unknown); located near Dock 131
- AOC #15: Area C – Two Former Fuel Oil ASTs (size unknown); located near Former Building 417
- AOC #16: Area D – Dumping Area
- AOC #17: Area E – Former Coal Trestle and Storage Area; located near Former Building 410
- AOC #18: Area F – Former Coal Storage Area; located near Former Building 215

[2] The ASTs themselves are not the AOCs. The FUDS-eligible AOCs are the former AST locations/footprints. The ASTs were removed but the sites were not formally closed and were not included in the CONHTRW project.

- AOC #19: Three Former Transformers – (T1 northwest of Building 406, T2 near AOC #16 – Area D [Dumping Area], and T3 northeast of Building 220).

The former buildings/structures and foundations were demolished/removed by the Palisades Interstate Park Commission (PIPC) between 1965 and 1972 (U.S. Army Geospatial Center [USAGC] 2018). The AOCs have been identified based on historical documentation/ records, historical drawings, and aerial photographs that were georeferenced within geographic information system (GIS) software. The preliminary contaminants of potential concern (COPCs) identified for the Iona Island Naval Ammunition Depot FUDS vary based on prior usage of former buildings/structures. A comprehensive list of preliminary COPCs identified for this HTRW RI include target analyte list (TAL) metals, PAHs, volatile organic compounds (benzene, toluene, ethylbenzene, and xylenes [BTEX], polychlorinated biphenyls (PCBs), and explosives residues. **Table 1** provides a brief summary of COPCs by AOC.

Previous investigations indicated that COPCs were likely mobilized beyond the footprint of the 19 AOCs during demolition of former buildings/structures and reworking of surface and subsurface soil. Those areas where COPCs were previously detected in surface and/or subsurface soil at concentrations above applicable screening levels[1] were included for further evaluation in the HTRW RI.

In addition to the above listed AOCs, a review of additional former buildings was conducted during preparation of the UFP-QAPP at the request of the U.S. Army Corps of Engineers (USACE) to identify locations where filling or possible spilling of explosive-containing materials could have occurred (**Table 2**). Seven former explosive storage and filling operations buildings were identified as having significant potential for explosive hazard based on limited information available in previous reports and input from USACE (**Figure 2**). Information regarding utilities, piping, building sumps, or other additional release pathways for explosives at these locations is not available. The buildings have been demolished and associated slabs removed, and the underlying soil has been reworked. Although these buildings were not included in the C02NY074402 HTRW project, the presence of explosives material in soil at these former building locations has not been previously evaluated. Therefore, these areas (listed below) were evaluated during the Phase I investigation at the request of USACE to confirm the presence/absence of explosives constituents in soil and to evaluate potential exposure risk from remaining explosives residues to human and ecological receptors.

- Building 609 – Mine Loading Plant
- Building 506 – Filling House #3 (Bombproof)
- Building 503 – Filling House #6
- Building 404 – Filling House #1
- Building 307 – Filling House #5
- Building 306 – Filling House #2
- Building 202 – Shell House #5 & D Plant.

The HTRW RI is the first comprehensive investigation of the Iona Island Naval Ammunition Depot FUDS. A two-phased approach is being employed to facilitate data collection, evaluation,

and discussion. The Phase I investigation was conducted to establish background soil concentrations for metals and PAHs, evaluate the presence/absence and/or nature and extent of COPCs in surface soil (from 0-6 inches [in.] below ground surface [bgs]) and shallow subsurface soil (from 6-36 in. bgs or refusal), and evaluate potential risk to human and ecological receptors.

A shoreline reconnaissance survey was also conducted as part of Phase I (April 6-7, 2020) to identify potential pathways (i.e., channels, drainage ditches, culverts, sewage outfalls/discharge pipes, etc.) that lead to the shoreline environment, evaluate whether there are barriers to overland transport from Iona Island to the shoreline (e.g., the railroad), and conduct a receptor and resource inventory evaluation. The shoreline reconnaissance is discussed in a separate technical memorandum that was submitted in April 2020 (EA 2020b). Seven sediment samples were collected as part of the shoreline reconnaissance for analysis of grain size and total organic carbon (TOC) to identify physical characteristics of sediment and evaluate whether sediments could be a potential "sink" for site-related contaminants (**Figure 3**). Analytical results will be used to evaluate the potential transport of COPCs from the FUDS to the shoreline and the subsequent risk to human and ecological receptors as well as assist in the design of the Phase II investigation (if needed). Only the Phase I ISM data will be used in the quantitative human health risk assessment. Discrete data will be used to determine if more and unbiased samples need to be collected to support determining 95% UCLs for chemicals in media that were sampled to determine presence/absence of COCs (e.g., subsurface soil for future construction worker).

The Phase II investigation, including media, sampling locations, and analytes to be sampled, are contingent upon the results of the Phase I investigation. Phase II activities may include the following:

- Collection of additional samples and data which meet the DQO input for quantitative risk assessments.
- Additional sampling of onsite surface soil and/or shallow and/or deep subsurface soil as necessary to refine impacts to these environmental media at any given AOC/area.
- Supplemental sampling of other environmental media (i.e., sediment, surface water, and/or overburden, and bedrock groundwater) as necessary.
- Toxicity testing (i.e., tissue sampling, toxicity testing, and/or benthic community metrics) to further support development of the Baseline Ecological Risk Assessment.

Phase I Investigation Area

The Phase I investigation area encompassed the 19 AOCs eligible for further inclusion in the C02NY074402 HTRW project, previous surface and subsurface soil sampling locations where COPCs were detected at concentrations above applicable screening levels, and the seven former buildings identified as having a significant potential for explosive hazard. In addition, eight non-contiguous background sampling areas were identified within Bear Mountain State Park outside the influence of the Iona Island Naval Ammunition Depot FUDS (**Figure 4**). Background

sampling was included in the Phase I investigation to establish regional background metal and PAH concentration ranges in surface and subsurface soil. Background locations were selected from areas outside the influence of the Iona Island Naval Ammunition Depot FUDS and from areas with the same soil unit mapped at Iona Island (Chatfield rock outcrop complex) (Soil Survey Staff, Natural Resources Conservation Service, United States Department of Agriculture 2018). Chatfield-Rock outcrop is the primary native soil at Iona Island and is classified as well-drained gravelly-sandy loam soil, 20 to 40 in. deep to the restrictive feature. Background locations were refined during the Phase I field investigation based on conditions encountered in the field (presence of exposed bedrock, soil color, texture, size and shape of soil aggregates, kind and amount of rock fragments, soil organic content, etc.). Locations were moved as needed based on observations in the field to ensure consistency between background and onsite locations.

Incremental Sampling Methodology Sampling Design

An incremental sampling methodology (ISM) approach was selected for the Phase I investigation to reduce data variability and increase sample representativeness. Decision units (DUs) were established for onsite and background sampling locations following the procedures in the UFP-QAPP (EA 2020a). A total of 25 onsite surface and subsurface soil DUs were established to address HTRW AOCs and locations where elevated concentrations of COPCs were detected during previous investigations. The 25 DUs included 19 DUs sized at approximately 1 acre (200 x 200 feet [ft]) (DU-1 through DU-19) and 6 smaller DUs sized at 60 x 60 ft (DU-20 through DU-25) (**Figure 5**). An additional seven DUs approximately 1-acre in size (200 x 200 ft or 100 x 400 ft) were established at former buildings identified as having a significant potential for explosive hazard (EXDU-1 through EXDU-7). A total of 8 offsite background DUs were established in Bear Mountain State Park (BADU-1 through BADU-8) (**Figure 4**).

DUs were modified at the start of the Phase I field investigation based on conditions encountered in the field (i.e., presence of exposed bedrock limiting soil sample collection). Separate surface soil (0-6 in. bgs) and shallow subsurface soil (6-36 in. bgs) incremental samples were proposed to be collected within each onsite DU established to address HTRW AOCs and locations where elevated concentrations of COPCs were detected during previous investigations (DU-1 through DU-25), and within each background DU (BADU-1 through BADU-8) to investigate potential risk to ecological and human receptors. Separate surface soil (0-6 in. bgs) incremental samples were proposed to be collected within each onsite DU established at former buildings identified as having a significant potential for explosive hazard (EXDU-1 through EXDU-7).

The analytes to be sampled at each DU are presented below and were based on previous investigation results and COPCs identified for each AOC (**Table 1 and 2**).

- TAL metals (excluding mercury) by EPA Method 6010C/6020A in surface and subsurface soil from:
    - DU-1 through DU-19, and
    - BADU-1 through BADU-8.

- Mercury by EPA Method 7471B in surface and subsurface soil from:
    - DU-1 through DU-5,

  - DU-8 and DU-9,
  - DU-11 through DU-19, and
  - BADU-1 through BADU-8.

- PAHs by EPA Method 8270D selected ion monitoring from surface and subsurface soil from:
  - DU-1 through DU-5,
  - DU-8 and DU-9,
  - DU-11 through DU-13,
  - DU-16 through DU-19,
  - DU-20 through DU-22, and
  - BADU-1 through BADU-8.

- PCBs by EPA Method 8082A from surface and subsurface soil from:
  - DU-17 through DU-19, and
  - DU-23 through DU-25.

- Explosives residues by Method 8330B from surface and subsurface soil from:
  - DU-17 through DU-19, and
  - EXDU-1 through EXDU-7.

- BTEX by EPA Method 8260B from subsurface soil only from:
  - DU-20 through DU-22.

- pH by field screening (surface and subsurface soil at all onsite and background DUs).

Pre-Investigation Field Activities

Equipment and supplies for the Phase I investigation were transported to the site on 9 March 2020. Pre-investigation field activities including a munitions and explosives of concern (MEC) surface sweep, brush / vegetation reduction, and flagging of DU grid corners, were conducted from 9 to 13 March 2020. Photographs of the key field activities are presented in **Attachment 1** and the location of each photograph was mapped on **Figure 6**.

Incremental Sampling Methodology Sample Collection

Phase I ISM sampling was conducted from 16 to 20 March and 30 March through 1 April 2020. Samples at each onsite DU were collected in triplicate for the purpose of calculating the 95 percent upper confidence limit of the mean using Chebychev statistical tests. Single samples were collected from each of the eight background DUs. Sample collection information for each DU is presented in **Table 3** and discussed in the subsections below.

Incremental soil samples were prepared by collecting multiple increments of soil within a DU and physically combining these increments into a single sample, referred to as the "incremental sample." Each incremental sample collected from each approximately 1-acre (200 x 200 or 100 x

400 ft) sized DU (DU-1 through DU-19, EXDU-1 through EXU-7, and BADU-1 through BADU-8) was comprised of 50 increments of soil, while each incremental sample collected from each smaller 60 x 60 ft DU (DU-20 through DU-25) was comprised of 30 increments of soil.

Surface soil ISM samples (0-6 in. bgs) were collected from each onsite and background DU using random sampling within each grid pattern, while avoiding concrete, exposed bedrock, pavement, shallow depth to refusal, and other features that inhibited collection of a surface soil increment. Surface soil increments were collected from each grid square using a dedicated incremental sampling tool with plungers collecting soil approximately equivalent to a specified volume of soil. The depth of each increment of soil was approximately 6 in., which was the length of the incremental sampling tool's sampling plunger attachment. Each of the increments of soil for the 200 x 200 ft DUs (50 increments for 1 ISM sample) was approximately 20 grams, to provide a total weight of 1 kilogram of soil. Each of the increments of soil for the 60 x 60 ft DUs (30 increments for 1 ISM sample) was approximately 33 grams, to provide a total weight of 1 kilogram of soil. Increments for each analysis were placed into 5-gallon buckets with disposable bucket liners. Upon completion of the desired number of increments, the increments were combined into a single sample, referred to as the "incremental sample."

Shallow subsurface soil samples (6-36 in. bgs) were collected from randomly located hand-auger soil borings advanced in DUs where soil depth was greater than 12 in. bgs. Subsurface soil depths were initially evaluated when attempting to collect surface soil increments. Following completion of surface soil sampling, a minimum of 5 borings were advanced in each DU to evaluate subsurface soil conditions. Boring locations were advanced in areas where there was no evidence of bedrock or hardscapes. Surface and shallow subsurface soil conditions (i.e., exposed bedrock, depth to refusal) were documented in the field logbook. Subsurface soil was only encountered at five onsite DUs (DU-17 through DU-21). At the remaining onsite and background DUs, shallow subsurface soil was limited to 12 in. bgs and, therefore, no subsurface soil samples were collected from these DUs.

For the subsurface soil samples, individual soil increments were collected from each soil boring, producing a total of 30 subsurface soil increments for each 60 ft x 60 ft DU and 50 subsurface soil increments for each of the approximately 1-acre sized DUs.

Subsamples for TAL metals, mercury, PAHs, PCBs, and explosives residue analyses were taken from the surface or subsurface incremental soil sample in the analytical laboratory. Samples for BTEX analysis were collected in the field using Terra Core samplers and placed in laboratory-preserved jars containing methanol.

pH for surface and subsurface soil samples was determined in the field using a pH meter in accordance with EPA Method 9045D "Soil and Waste pH." pH measurements were recorded in the field book. Soil geological characteristics for each DU including color, moisture, texture, grain size, sorting, lithology, etc. were recorded in the field book.

DU-1

DU-1 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #17 – Area E (Former Coal Trestle and Storage Area) and AOC #10 – Former Building 406 (Tin, Electrical and Annealing Shop Building). Surface soil samples were collected in triplicate on 18 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the presence of shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal ranged from 6 to 8 in. bgs.

Surface soil was characterized as brown, dry silt and organic material with trace fine- to coarse-grained sand and gravel (OL) (American Society for Testing and Materials [ASTM] International 2006). Recorded pH values for triplicate surface soil samples were 5.6, 6.5, and 6.8.

DU-2

DU-2 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #17 – Area E (Former Coal Trestle and Storage Area). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the presence of exposed and shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal ranged from 6 to 8 in. bgs.

Surface soil was characterized as brown, dry silt and organic material with trace fine- to coarse-grained sand and gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 5.6, 6.6, and 6.8.

DU-3

DU-3 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #12 – Former Building 410 (Power House) and AOC #11 – Former Building 407 (Paint Shop and Pipe Shop). Surface soil samples were collected in triplicate on 18 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the presence of exposed and shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown, dry silt with organics and trace fine- to coarse-grained sand and gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.0, 7.0, and 7.1.

DU-4

DU-4 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at previous sampling location II-EA-SD-02-01 (which had elevated concentrations of antimony, copper, lead, and mercury) and previous sampling location IX407-B1 (which had elevated concentrations of arsenic, lead, and fluoranthene) (Figure 5). Surface soil samples were collected in triplicate on 18 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the presence of significant anomalies (metal debris) and shallow bedrock and shallow depth to refusal throughout the DU. Six hand auger borings were advanced to evaluate soil depth. Soil depth to refusal at one boring was 12 in. bgs, while soil depth to refusal at the remaining borings ranged from 4 to 8 in. bgs.

Surface soil was characterized as brown, dry silt with organics and trace fine- to coarse-grained sand and gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.9, 7.0, and 7.1.

DU-5

DU-5 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at AOC #3 – Former Building 123 (Garage Building) and previous sampling location II-EA-SS-02-06 (which had elevated concentrations of antimony, copper, lead, mercury, and zinc). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the presence of exposed and shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown, dry silt with organics and trace fine- to coarse-grained sand and gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were all 7.1.

DU-6

DU-6 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of previous sampling locations II-EA-SS-02-09 (which had an elevated concentration of copper) and EA-SS-02-03 (which had elevated concentration of lead). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals excluding mercury. Subsurface soil samples were not collected due to the presence of shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown with some light brown and yellow coloring, slightly moist silt with trace clay and very fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were all 7.5.

DU-7

DU-7 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of previous sampling location II-EA-SS-02-08 (which had elevated concentrations of copper, lead, and zinc). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals excluding mercury. Subsurface soil samples were not collected due to the presence of shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown, dry silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.4, 7.4, and 7.5.

DU-8

DU-8 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #5 – Former Building 207 (Marine Garage Building). The DU is contained by existing buildings 201, 212, and 222. Numerous rusted and deteriorating 55-gallon drums, deteriorating pallets, piles of roofing shingles, chain link fencing, and other debris were observed in DU-8 during the mark-out the week of 9 March 2020 and a site reconnaissance conducted on 11 March 2020. Several empty 55-gallon drums were lying on their sides, while other drums were upright and filled with fluid. The material contained within the drums is not known. Photographs of conditions at DU-8 are presented in **Attachment 1**. Based on photographs of the area taken in 1965 just before the property was turned over to PIPC, these drums and debris are not attributable to past DoD activities (USAGC 2018).

Per discussion with USACE on 24 March 2020, it was concluded that surface and subsurface soil samples would not be collected from DU-8, as it would not be possible to separate metals and PAH contaminants originating from former DoD operations at Building 207 from the contaminants originating within the drums and other debris observed in the DU. New York State Department of Environmental Conservation (NYSDEC) was notified by USACE on 2 April 2020 that DU-8 would not be sampled.

DU-9

DU-9 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #8 – Former Building 219 (Garage Building) and AOC #9 – Former Building 220 (Garage Buildings). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the shallow depth to bedrock and shallow depth to refusal throughout the DU. Ten hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.1, 6.9, and 7.0.

DU-10

DU-10 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity previous sampling location II-EA-SS-02-14 (which had elevated concentration of copper). Surface soil samples were collected in triplicate on 19 March 2020 for analysis of TAL metals excluding mercury. Subsurface soil samples were not collected due to the presence of shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.1, 7.1, and 7.2.

DU-11

DU-11 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #18 – Area F (Former Coal Storage Area). Surface soil samples were collected in triplicate on 30 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 2 in.

Surface soil was characterized as brown, moist, well sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.0, 7.0, and 7.2.

DU-12

DU-12 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #6 – Former Building 213 (Paint Locker for Building 202) and AOC #7 – Former Building 215 (Garage Building). Surface soil samples were collected in triplicate on 30 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 2 in.

Surface soil was characterized as dark brown, moist, well sorted silt and organic material with trace fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 5.5, 6.1, and 6.3.

DU-13

DU-13 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #1 – Former Building 103 (Paint and Oil Storage) and previous sampling location IX-103-B1 (which had elevated concentrations of arsenic, cadmium, lead, fluoranthene, and pyrene). Surface soil samples were collected in triplicate on 30 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 2 in.

Surface soil was characterized as dark brown, moist, well sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.8, 7.0, and 6.9.

DU-14

DU-14 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of previous sampling location II-EA-SS-02-13 (which had elevated concentrations of lead and mercury). Surface soil samples were collected in triplicate on 20 March 2020 for analysis of TAL metals including mercury. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as dark brown, dry, well sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.7, 6.9, and 7.0.

DU-15

DU-15 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of previous sampling location II-EA-SS-02-15 (which had elevated concentrations of lead and mercury). Surface soil samples were collected in triplicate on 1 April 2020 for analysis of TAL metals including mercury. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 12 in.

Surface soil was characterized as dark brown, dry, well sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.2, 6.3, and 6.6.

DU-16

DU-16 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #4 – Former Buildings 124-128 (Sewage Disposal Plant). Surface soil samples were collected in triplicate on 31 March 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 12 in.
Surface soil was characterized as dark brown, dry, well sorted silt and organic material with trace fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.9, 6.9, and 7.4.

DU-17

DU-17 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at and in the vicinity of AOC #2 – Former Building 121 (Incinerator), AOC #16 – Area D (Dumping Area), and previous sampling locations IX-121-B1 (which had elevated concentrations of arsenic, cadmium, lead, fluoranthene, and pyrene) and II-EA-SD-02-02 (which had elevated concentrations of antimony, copper, lead, nickel, and zinc). Surface and subsurface soil samples were collected in triplicate on 31 March 2020 for analysis of TAL metals including mercury, PAHs, Aroclor PCBs, and explosives residues.

Surface soil was characterized as dark brown, moist, well sorted silt and organic material with trace fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.2, 7.2, and 7.1.

Subsurface soil was characterized as black, very moist, poorly sorted sand (SW) (ASTM 2000). Depth to refusal was greater than 3 ft. Recorded pH values for triplicate subsurface soil samples were 7.0, 7.4, and 7.7.

DU-18

DU-18 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at AOC #16 – Area D (Dumping Area) and in the vicinity of previous sampling locations IX DMP-B22 (which had elevated concentrations of arsenic, cadmium, chromium, lead, silver, 2,4-dinitrotoluene [DNT], 2,6-DNT, and Aroclor 1260) and IXDMP-B1 (which had elevated concentrations of lead and Aroclor 1260). Surface and subsurface soil samples were collected in triplicate on 31 March 2020 for analysis of TAL metals including mercury, PAHs, Aroclor PCBs, and explosives residues.

Surface soil was characterized as dark brown, moist, moderately sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.5, 8.0, and 7.8.

Subsurface soil was characterized as very moist, dark brown to grey silt with trace fine-grained sand (ML) (ASTM International 2006). Depth to refusal was greater than 3 ft. Recorded pH values for triplicate subsurface soil samples were 8.1, 8.1, and 8.0.

DU-19

DU-19 is a 200 x 200 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at AOC #16 – Area D (Dumping Area) and in the vicinity of previous sampling location II-EA-SS-02-16 (which had elevated concentrations of antimony, copper, lead, mercury, and zinc). Surface and subsurface soil samples were collected in triplicate on 31 March 2020 for analysis of TAL metals including mercury, PAHs, Aroclor PCBs, and explosives residues.

Surface soil was characterized as dark brown, moist, moderately sorted silt and organic material with trace fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.7, 7.5, and 7.4.

Subsurface soil was characterized as very moist, dark grey (with a bluish tint), well sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Depth to refusal was greater than 3 ft. Recorded pH values for triplicate subsurface soil samples were 7.0, 7.4, and 7.7.

DU-20

DU-20 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at a former AST located near former Building 233 (AOC #13 – Area A). Surface soil samples were collected in triplicate on 16 March 2020 for analysis of PAHs. Subsurface soil samples were collected in triplicate on 17 March 2020 for analysis of PAHs and BTEX.

Surface soil was characterized as dry, brown, poorly sorted silt with trace fine- to coarse-grained sand and fine to medium gravel (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.1, 6.5, and 6.6.

Subsurface soil was characterized as dry, brown, poorly sorted silt with trace fine- to coarse-grained sand and fine to medium gravel (ML) (ASTM International 2006). Depth to refusal was approximately 18 in. Recorded pH values for triplicate subsurface soil samples were 6.8, 6.9, and 6.8.

DU-21

DU-21 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at a former AST located near former Dock 131 (AOC #14 – Area B). Surface soil samples were collected in triplicate on 17 March 2020 for analysis of PAHs. Subsurface soil samples were collected in triplicate on 16 March 2020 for analysis of PAHs and BTEX.

Surface soil was characterized as dry, brown, poorly sorted silt with trace fine- to coarse-grained sand and fine- to coarse-grained gravel (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.3, 6.8, and 6.8.

Subsurface soil was characterized as dry, brown, poorly sorted silt with trace fine- to coarse-grained sand and fine to medium gravel (ML) (ASTM International 2006). Depth to refusal was approximately 24 in. Recorded pH values for triplicate subsurface soil samples were 7.0, 7.1, and 7.3.

DU-22

DU-22 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at two former ASTs located near former Building 407 (AOC #15 – Area C). Surface soil samples were collected in triplicate on 16 March 2020 for analysis of PAHs. Subsurface soil samples were not collected due to the shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as dry, brown, poorly sorted silt with trace fine- to coarse-grained sand and fine- to coarse-grained gravel (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.7, 6.8, and 6.8.

DU-23

DU-23 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at former transformer T1 (AOC #19). Surface soil samples were collected in triplicate on 20 March 2020 for analysis of Aroclor PCBs. Subsurface soil samples were not collected due to the presence of exposed bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 6 in.

Surface soil was characterized as dry, dark brown, moderately sorted silt with trace fine-grained sand (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.0, 5.4, and 5.5.

DU-24

DU-24 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at former transformer T2 (AOC #19). DU-24 is bounded by the Hudson River to the east/northeast and the former dump area between Iona and Round Islands to the northwest. Historical documents indicated that former transformers on a pole were located along the former eastern sentry path from Iona Island onto Round Island. A downed former electric pole was noted during a reconnaissance on 20 March 2020.

The majority of DU-24 consists of exposed bedrock with little thin soil cover. Photographs of conditions at DU-24 are presented in **Attachment 1**. Sediment and soil were noted along the

northeastern edge of DU-24 off the bedrock outcrop of Round Island; however, this sediment/soil was along the shoreline of the tidally influenced Hudson River. There was little to no soil available for sampling in the area of the former transformer. Any PCBs that may have been released from former transformers would have been transported via runoff and overland flow across the bedrock outcrop of Round Island to the Hudson River. However, possible PCB impacts to the Hudson River from the Site would be indistinguishable from documented upriver PCB sources.

Per discussion with USACE on 24 March 2020, it was concluded that surface and subsurface soil samples would not be collected from DU-24 due to the lack of soil in the area of the former transformer and the Hudson River's influence on sediment/soil along the shoreline. NYSDEC was notified by USACE on 2 April 2020 that DU-24 would not be sampled.

DU-25

DU-25 is a 60 x 60 ft DU established to evaluate potential contamination in surface soil (0-6 in. bgs) and subsurface soil (6-36 in. bgs) at former transformer T3 (AOC #19). Surface soil samples were collected in triplicate on 20 March 2020 for analysis of Aroclor PCBs. Subsurface soil samples were not collected due to the presence of exposed bedrock/a bedrock wall and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 8 in.

Surface soil was characterized as dry, brown, moderately sorted silt with organic material (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.2, 6.4, and 6.4.

EXDU-1

EXDU-1 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 609 – Mine Loading Plant. Surface soil samples were collected in triplicate on 17 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, dry silt with trace fine- to coarse-grained sand and gravel (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.0, 6.9, and 6.9.

EXDU-2

EXDU-2 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 506 – Filling House #3. Surface soil samples were collected in triplicate on 17 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, dry silt with trace fine- to coarse-grained sand and gravel (ML) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.0, 6.8, and 6.9

EXDU-3

EXDU-3 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 503 – Filling House #6. Surface soil samples were collected in triplicate on 18 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, slightly moist silt and organics with trace clay and very fine to fine-grained sand (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.3, 6.5, and 5.5.

EXDU-4

EXDU-4 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 404 – Filling House #1. Surface soil samples were collected in triplicate on 17 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, dry silt with trace fine- to coarse-grained sand and gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 6.5, 6.3, and 6.8.

EXDU-5

EXDU-5 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 307 – Filling House #5. Surface soil samples were collected in triplicate on 18 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, slightly moist silt with organics and trace clay (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.1, 7.2, and 7.3.

EXDU-6

EXDU-6 is a 200 x 200 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 306 – Filling House #2. Surface soil samples were collected in triplicate on 18 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, slightly moist silt with organics and trace clay and coarse gravel (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.2, 7.3, and 7.3.

EXDU-7

EXDU-7 is a 100 x 400 ft DU established to evaluate the presence or absence of residual explosives contamination in surface soil from Building 202 – Shell House #5 and D Plant. Surface soil samples were collected in triplicate on 18 March 2020 for analysis of explosives residues.

Surface soil was characterized as brown, slightly moist silt with organics and trace very fine sand and clay (OL) (ASTM International 2006). Recorded pH values for triplicate surface soil samples were 7.0, 7.0, and 7.1.

BADU-1

BADU-1 is a 200 x 200 ft DU located in Bear Mountain State Park northwest of the intersection of Perkins Memorial Drive and Seven Lakes Drive. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was less than 12 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). The pH of surface soil was recorded as 6.3.

BADU-2

BADU-2 is a 200 x 200 ft DU located in Bear Mountain State Park north of the intersection of Perkins Memorial Drive and Seven Lakes Drive. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). The pH of surface soil was recorded as 6.7.

BADU-3

BADU-3 is a 200 x 200 ft DU located in Bear Mountain State Park south of the intersection of Perkins Memorial Drive and Seven Lakes Drive. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). The pH of surface soil was recorded as 6.0.

BADU-4

BADU-4 is a 200 x 200 ft DU located in Bear Mountain State Park southeast of the intersection of Perkins Memorial Drive and Seven Lakes Drive. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout

the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). The pH of surface soil was recorded as 6.1.

BADU-5

BADU-5 is a 200 x 200 ft DU located in Bear Mountain State Park north of Seven Lakes Drive and west of Suffren Bear Mountain Trail. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). pH of surface soil was recorded as 6.1.

BADU-6

BADU-6 is a 200 x 200 ft DU located in Bear Mountain State Park at the intersection of Suffren Bear Mountain Trail and Seven Lakes Drive. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). pH of surface soil was recorded as 5.9.

BADU-7

BADU-7 is a 200 x 200 ft DU located in Bear Mountain State Park south of Seven Lakes Drive and southwest of the Suffren Bear Mountain Trail intersection. A surface soil ISM sample was collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). pH of surface soil was recorded as 6.1.

BADU-8

BADU-8 is a 200 x 200 ft DU located in Bear Mountain State Park south of Seven Lakes Drive and southeast of the Suffren Bear Mountain Trail intersection. A surface soil ISM sample was

collected on 1 April 2020 for analysis of TAL metals including mercury, and PAHs. Subsurface soil was not collected due to the presence of exposed/shallow bedrock and shallow depth to refusal throughout the DU. Five hand auger borings were advanced to evaluate soil depth, and the soil depth to refusal was between 3 and 8 in.

Surface soil was characterized as brown, slightly moist silt with organics, trace clay and very fine-grained sand (OL) (ASTM International 2006). The pH of surface soil was recorded as 6.0.

Sample Labeling and Shipping

Each ISM sample was assigned a unique alpha-numeric code to assist in the tracking of samples and to facilitate retrieval of analytical results. Sample bags/containers were labeled with weatherproof sample labels and waterproof ink containing the following information:

- Sample identification
- Sampling date and time
- Name of sampler
- Analysis requested
- Preservation technique.

Samples were placed on ice in sample coolers immediately after collection to ensure proper preservation. A temperature blank was included with samples as required to document laboratory receipt temperature. Each cooler was cleaned, lined with bubble wrap, and double-bagged ice was packed around the samples. Samples were packaged and shipped in a manner that ensured the safety and accountability of each sample, and the procedures implemented were in accordance with applicable federal and local requirements. A completed chain-of-custody form was placed in a sealed plastic bag and taped to the inside lid of one of the coolers for each shipment. Coolers were sealed with tape and a custody seal was attached to protect the integrity of the samples. Environmental samples were then shipped to the analytical laboratory via overnight delivery service.

Laboratory Analysis

Laboratory analytical services were provided by TestAmerica, a DoD certified laboratory. BTEX samples were shipped directly to TestAmerica Denver for processing and analysis. Grinding and air drying did not apply to the BTEX samples. PAHs, TAL metals, PCBs, and explosives samples were shipped to TestAmerica St Louis for processing (air drying and grinding). Ball mill grinding was utilized during laboratory sample processing of TAL metals, PAH, and PCB soil samples as some of the PAHs were likely to volatilize or degrade during puck mill grinding due to heat generation by the process. Puck mill grinding was utilized during laboratory processing of explosives soil samples. Following completion of sample processing, PAHs, TAL metals, PCBs, and explosives samples were shipped to TestAmerica Denver for analysis.

FUDSChem

Analytical data were managed in FUDSChem per the requirements specified in the New England District Data Management Plan to ensure data deliverables follow Performance Work Statement data requirements.

Prior to field sampling, an eQAPP within FUDSChem was created; the eQAPP was fully compliant with the project UFP-QAPP, incorporating methods, sample types, and quality specifications (limit of detection [LOD], limit of quantitation [LOQ], holding time, quality control [QC] sample acceptance limits, etc.). The pre-population of sampling event information was performed to ensure accurate database field matching and status tracking within FUDSChem. Data uploaded to FUDSChem prior to field sampling included spatial information pertaining to DUs (northing, easting, elevation of the center of each DU, and keyhole markup language [KML] files for each DU), sample identification information, sample type, matrix, methods, etc. Following completion of sampling, chain-of-custody documents (ChemFields) were entered within FUDSChem using pre-populated data and information contained in the eQAPP.

The analytical laboratories generated portable document format (PDF) laboratory reports and uploaded Stage 2A staged electronic data deliverables (SEDDs) to FUDSChem. Once a SEDD was successfully uploaded and certified by the analytical laboratory in FUDSChem, an Automated Data Review (ADR) process was automatically initiated. The ADR performed a Stage 2A data validation by comparing the SEDD to the approved eQAPP, summarizing QC outliers in an ADR Report, and applying data validation qualifiers to associated results. The EA data validator performed the Stage 2B data validation on the analytical data. The data validator reviewed the ADR Report against the PDF laboratory report to 1) verify/modify the ADR qualification of the sample results and to 2) supplement the ADR review with a manual review of QC elements that were not included in the ADR review. The data validator completed the data review checklist and prepared a data validation report that summarized the data validation findings. The EA chemist reviewed the data validation report and signed off as the second reviewer. The data validation report was uploaded to the FUDSChem library for USACE chemist approval.

Analytical Results

The validated analytical results and summary statistics are presented in the following tables:

- **Table 4**: DU-1 through DU-16
- **Table 5**: DU-17 through DU-19
- **Table 6**: DU-20 through DU-22
- **Table 7**: DU-23 and DU-25
- **Table 8**: EXDU-1 through EXDU-7
- **Table 9**: BADU-1 through BADU-8

Analytical results were compared to applicable EPA screening levels in accordance with CERCLA as follows:

- EPA residential and industrial Regional Screening Levels (RSLs) for soil (updated May 2020). Note that a deed restriction is in place at Iona Island noting that there is to be no residential development at the site (USACE 1995); however EPA Residential soil RSLs were included to assess for children receptors including site visitation for nature walks and potential camping.

- EPA Region 4 Ecological Soil Screening Levels (EcoSSLs) (March 2018) and screening values presented in Attachment 4 to the Ecological Risk Assessment Work Plan (Appendix E of the UFP-QAPP (EA 2020a).

Table 10 presents a list of analytes reported at concentrations exceeding screening criteria at each DU. BTEX was not reported at concentrations above screening criteria at former AST locations.

The following individual analytes were reported in soil samples at concentrations exceeding industrial soil RSLs:

- TAL metals (primarily arsenic, with select DUs with exceedances of lead, mercury, and copper)

- (primarily benzo[a]pyrene, with select locations with exceedances of benzo[a]anthracene, benzo[b]fluoranthene, and dibenz[a,h]anthracene)

The following individual analytes were reported in soil samples at concentrations exceeding EPA residential soil RSLs:

- TAL metals (primarily aluminum, arsenic, cobalt, iron, manganese, and thallium, with select DUs with exceedances of antimony, cadmium, copper, lead, mercury, vanadium, and zinc)

- PAHs (benzo[a]pyrene, benzo(a)anthracene, benzo[b]fluoranthene, dibenz[a,h]anthracene, and indeno(1,2,3-cd)pyrene)

- PCBs (total PCBs and PCB-1260 (Aroclor 1260) at AOC #16: Area D – Dumping Area only).

The following analytes were reported in soil at concentrations exceeding Eco SSLs:

- Metals (primarily antimony, arsenic, cadmium, copper, lead, manganese, mercury, selenium, thallium, vanadium, zinc, with select DUs with exceedances of barium, cobalt, chromium [total], and nickel)

- PAHs (benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, benzo[k]fluoranthene, chrysene, indeno[1,2,3-cd]pyrene, pyrene, total high molecular

weight PAHs, with select DUs with exceedances of dibenz[a,h]anthraene, phenanthrene, and total low molecular weight PAHs)

- PCBs (Total PCBs, PCB-1248 [Aroclor 1248], PCB-1260 [Aroclor 1260], and PCB-1262 [Aroclor 1262] at AOC #16: Area D – Dumping Area only) and Total PCBs and PCB-1260 (Aroclor 1260] at DU-23 only)

- Explosives (1,3-dinitrobenzene at EXDU-1 only).

Soil Background Comparison

Permutation test of means (Student t-Test with adjusted p-value) was used to compare onsite and background metals and PAH concentrations (**Table 11**). Permutation tests are a subset of nonparametric statistics. The two-sample permutation test of means is similar to a t-test in that it evaluates the difference between two group means, but in contrast to a t-test, the permutation test does not require that the data from each group follow a normal distribution or that the two groups have the same variance. For this reason, the permutation test is suitable for evaluating both small data sets and non-normal data distributions. Also, this permutation test does not require that the distribution of the two data groups have the same shape in order to be a central value test as does the commonly used Wilcoxon rank sum test.

The permutation test assumes only that the data from the two are "exchangeable" when the null hypothesis is true. For a two-sample permutation test of means, the test statistic is the observed difference in the two group means (i.e., site versus background). If the null hypothesis is true, the group assignment is arbitrary, as there is no difference in the means and the data in essence come from the same population. Therefore, the data are randomly rearranged regardless of group assignment to produce a different set of group means in each rearrangement. The difference in group means is computed and stored after each rearrangement, and this process is repeated for either all possible rearrangements (if possible) or several thousand permutations for larger data sets. The proportion of differences from the rearrangements that equal or exceed the one observed difference from the original data is the permutation p-value of the test (Helsel et al., 2020).

A comparison of analytes exceeding both screening criteria and background concentrations was conducted to evaluate whether concentrations were consistent with background or elevated above background concentrations. **Table 12** presents a list of analytes reported at concentrations exceeding both screening criteria and background concentrations at each DU.

The following metals and PAH analytes were reported in soil samples at concentrations exceeding both industrial soil RSLs and background concentrations:

- TAL metals (primarily arsenic, with select DUs with exceedances of lead, mercury, and copper)

- PAHs (primarily benzo[a]pyrene, with select locations with exceedances of benzo[a]anthracene, benzo[b]fluoranthene, and dibenz[a,h]anthracene)

The following metals and PAH analytes were reported in soil samples at concentrations exceeding EPA residential soil RSLs:

- TAL metals (primarily arsenic, cobalt, iron, manganese, and thallium, with select DUs with exceedances of aluminum, antimony, cadmium, copper, lead, mercury, vanadium, and zinc)

- PAHs (benzo[a]pyrene, benzo(a)anthracene, benzo[b]fluoranthene, dibenz[a,h]anthracene, and indeno(1,2,3-cd)pyrene)

- PCBs (total PCBs and PCB-1260 (Aroclor 1260) at AOC #16: Area D – Dumping Area only).

The following metals and PAH analytes were reported in soil at concentrations exceeding Eco SSLs:

- Metals (primarily arsenic, cadmium, copper, lead, manganese, and zinc with select DUs with exceedances of antimony, barium, cobalt, chromium [total], mercury, nickel, selenium, thallium, and zinc)

- PAHs (benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, benzo[k]fluoranthene, chrysene, indeno[1,2,3-cd]pyrene, pyrene, total high molecular weight PAHs, with select DUs with exceedances of dibenz[a,h]anthraene, phenanthrene, and total low molecular weight PAHs).

Sediment TOC and Grain Size

Grain size and TOC analytical results for sediment samples collected during the shoreline reconnaissance are presented in **Table 13**. This data will be used to evaluate potential background sediment locations during Phase II.

Discussion and Action Items

There were no analytes exceeding both screening criteria and background concentrations in surface soil at DU-25 and EXDU-2 through EXDU-7, and in surface and subsurface soil at DU-20 and DU-21. At each of the remaining DUs, one or more metals, PAH, PCB, and/or explosives analytes were reported at concentrations exceeding both screening criteria and background concentrations.

Evaluation of the Phase I ISM soil analytical data, including comparisons between onsite and background data and evaluations of risk to human health and ecological receptors, is ongoing. A human health risk assessment and a screening-level ecological risk assessment are being conducted

using the Phase I analytical data to evaluate whether constituent concentrations present a potential risk to human and terrestrial ecological receptors. Only the Phase I ISM data will be used in the quantitative human health risk assessment. Discrete data will be used to determine if more and unbiased samples need to be collected to support determining 95% UCLs for chemicals in media that were sampled to determine presence/absence of COCs (e.g., subsurface soil for future construction worker).

Analytical results and risk assessments will be presented in the Phase I Data Report, to be submitted following completion of data analysis and risk assessments. Results of the risk assessments will be used to determine the need for further investigation under Phase II. If results and risk assessments indicate that additional sampling is necessary to evaluate the nature and extent of contaminants, an addendum to the UFP-QAPP in the form of revised QAPP Work Sheets will be prepared to detail the Phase II data collection/investigation. The Phase II investigation design and rationale will be presented to the project delivery team in a Technical Project Planning Meeting (TPP #3) prior to conducting the Phase II field investigation.

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Attachments

A Photograph Log

FIGURES

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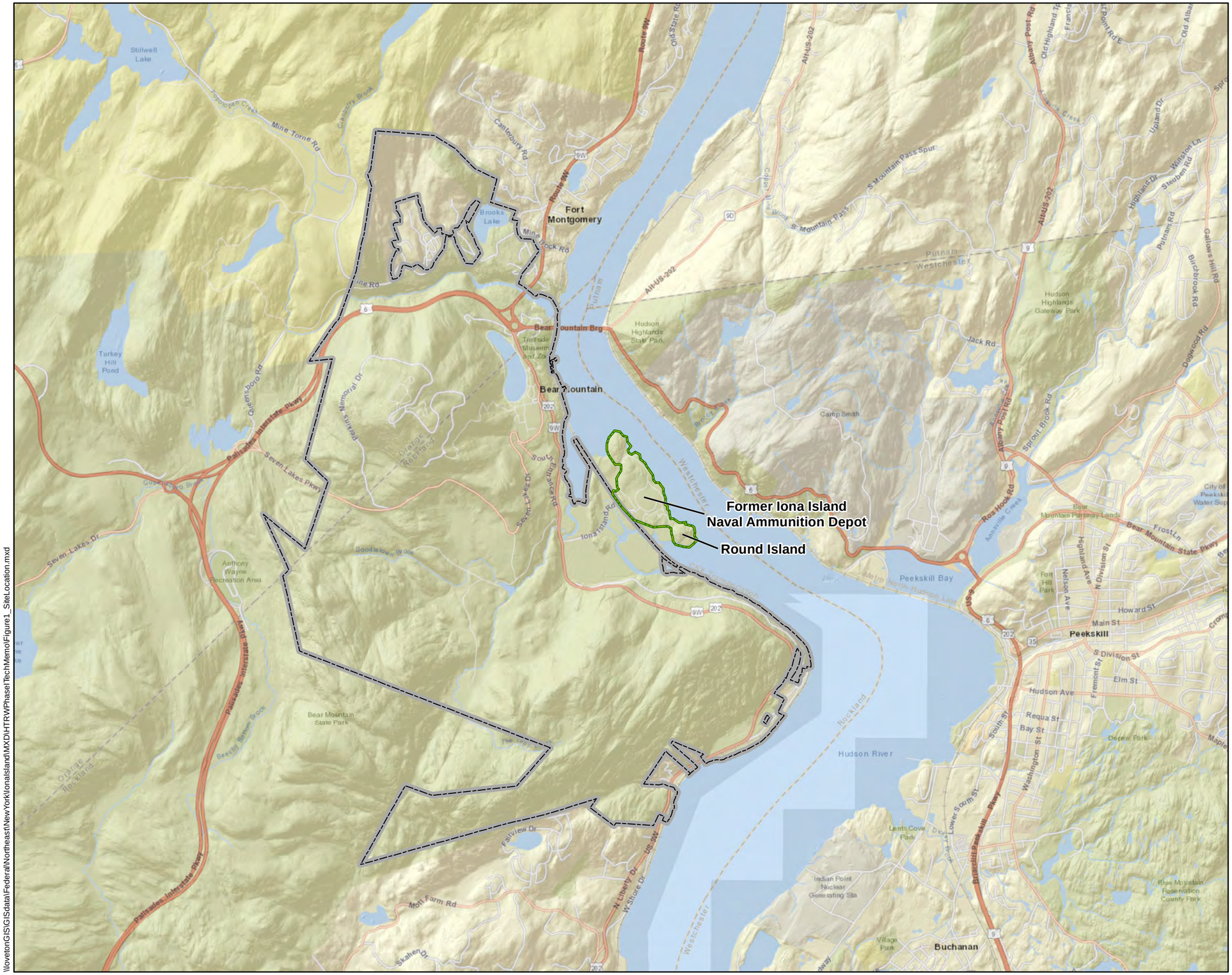
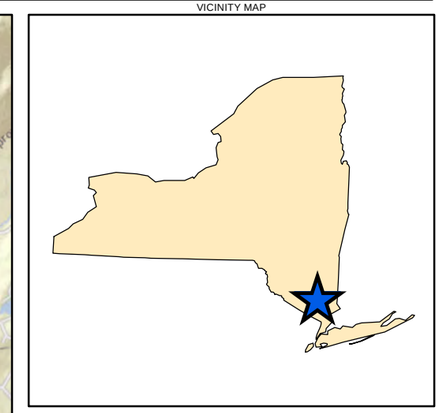


VICINITY MAP



Legend

- FUDS Boundary
- Bear Mountain State Park Boundary (NYS Office of Parks, Recreation & Historic Preservation, 2018)

0 0.5 1
Miles
1 in = 1 miles

N

FIGURE 1
General Location of the Iona Island Naval Ammunition Depot Formerly Used Defense Site

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Source
Basemap: ESRI World Street Map - ArcGIS Online Map Service (ESRI 2009).
DEM: 2012, Photo Science, Inc., Coastal New York LiDAR (Hydro Flattened Raster DEM), 1-Meter Resolution, New York State GIS

Map Date: 9/4/2020

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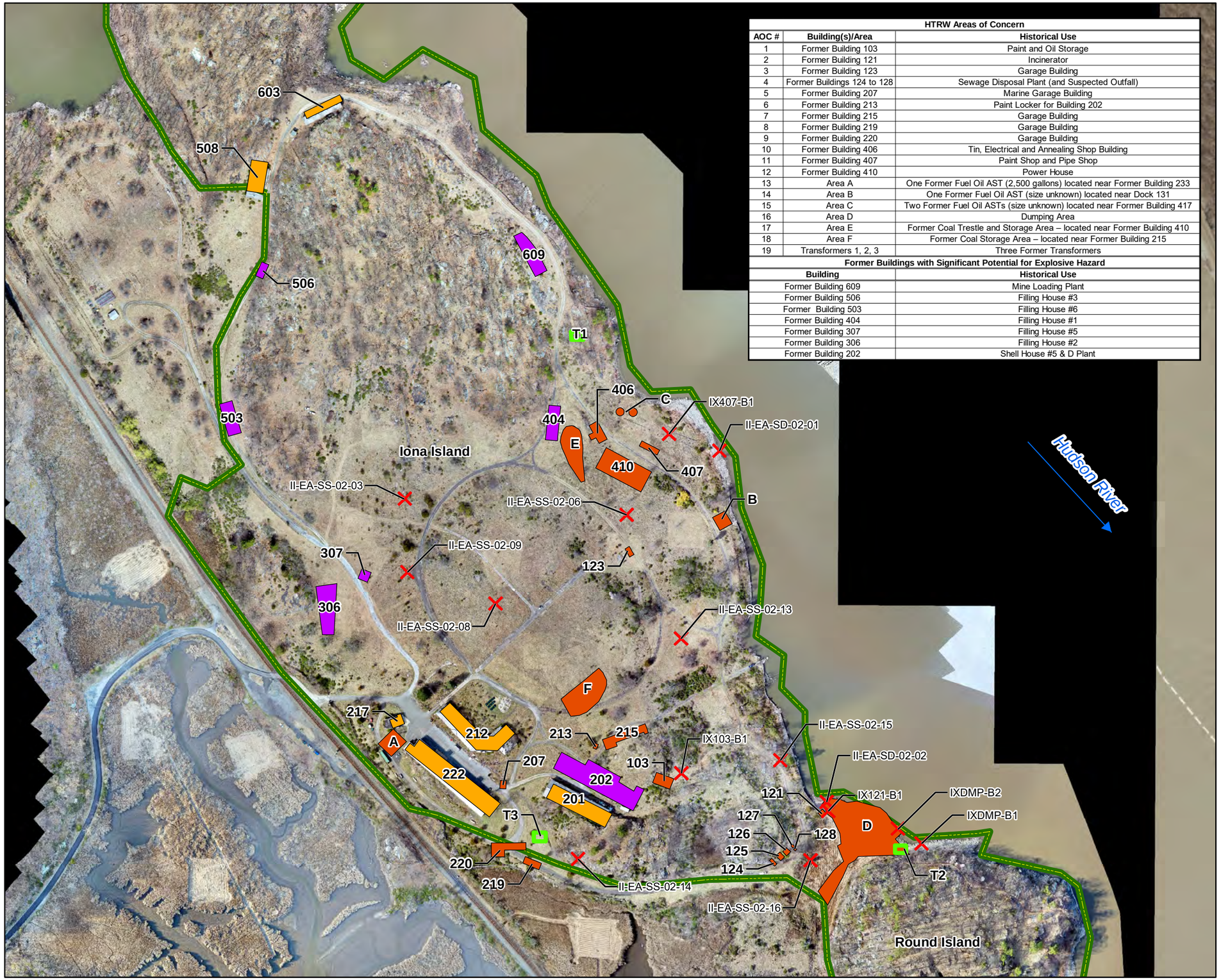

HTRW Areas of Concern		
AOC #	**Building(s)/Area**	**Historical Use**
1	Former Building 103	Paint and Oil Storage
2	Former Building 121	Incinerator
3	Former Building 123	Garage Building
4	Former Buildings 124 to 128	Sewage Disposal Plant (and Suspected Outfall)
5	Former Building 207	Marine Garage Building
6	Former Building 213	Paint Locker for Building 202
7	Former Building 215	Garage Building
8	Former Building 219	Garage Building
9	Former Building 220	Garage Building
10	Former Building 406	Tin, Electrical and Annealing Shop Building
11	Former Building 407	Paint Shop and Pipe Shop
12	Former Building 410	Power House
13	Area A	One Former Fuel Oil AST (2,500 gallons) located near Former Building 233
14	Area B	One Former Fuel Oil AST (size unknown) located near Dock 131
15	Area C	Two Former Fuel Oil ASTs (size unknown) located near Former Building 417
16	Area D	Dumping Area
17	Area E	Former Coal Trestle and Storage Area – located near Former Building 410
18	Area F	Former Coal Storage Area – located near Former Building 215
19	Transformers 1, 2, 3	Three Former Transformers

Former Buildings with Significant Potential for Explosive Hazard	
Building	**Historical Use**
Former Building 609	Mine Loading Plant
Former Building 506	Filling House #3
Former Building 503	Filling House #6
Former Building 404	Filling House #1
Former Building 307	Filling House #5
Former Building 306	Filling House #2
Former Building 202	Shell House #5 & D Plant

VICINITY MAP

Legend

- FUDS Boundary
- HTRW Project Areas of Concern
- FormerTransformers
- Remaining Structure (Not FUDS Eligible)
- Previous Elevated Concentration of COPC

0 300 600
Feet
1 in = 300 ft

N

FIGURE 2
HTRW Project Areas of Concern

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
PROJECT REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Source
Aerial: EA Engineering, Science, and Technology. April 7, 2020

Map Date: 8/21/2020

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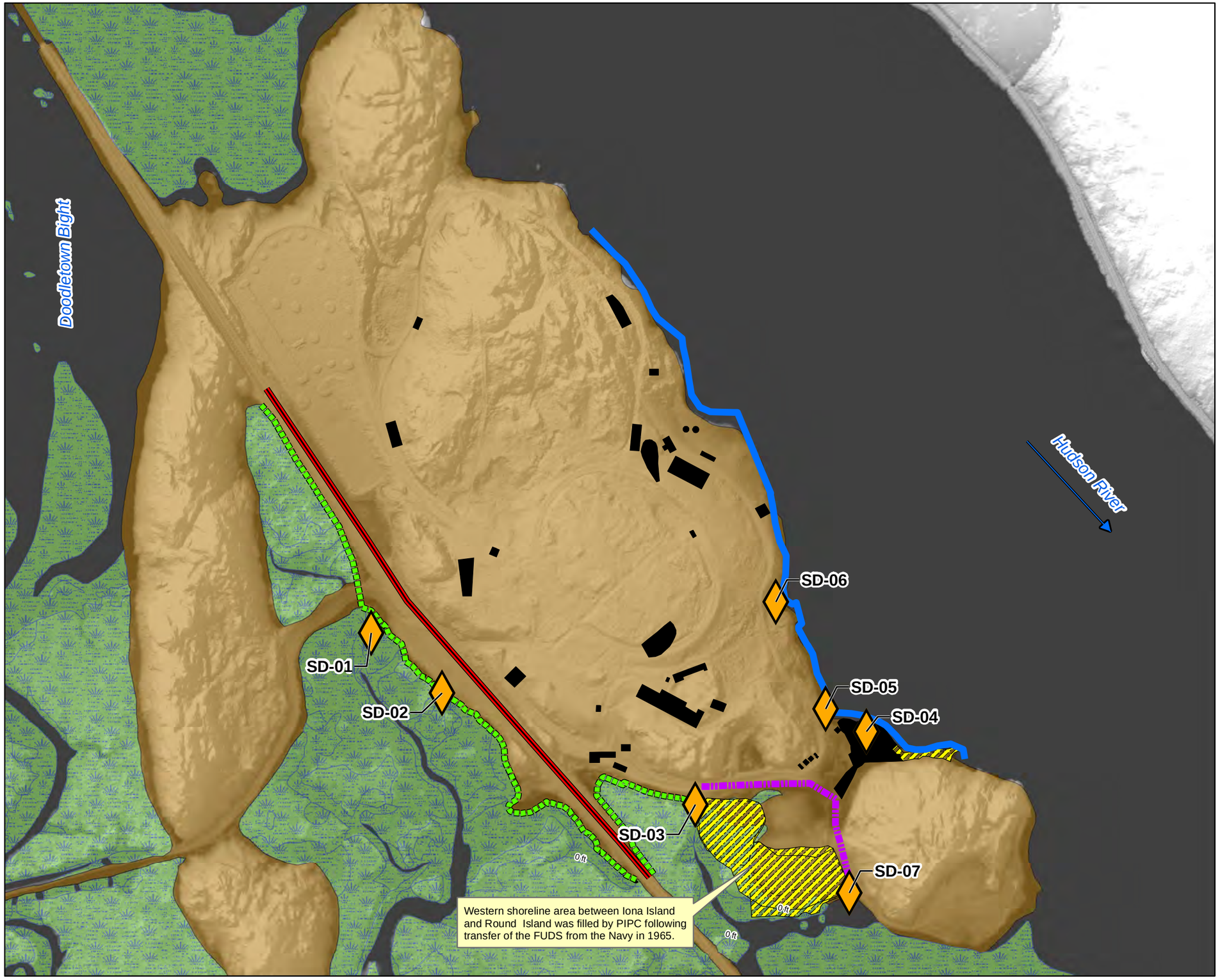


VICINITY MAP

Legend

- HTRW RI Footprints of Former Buildings/Structures
- Disturbed Ground/Fill Area
- Upland
- Marsh/Wetland

Phase I Shoreline Reconnaissance Areas

- Recon Area 1 Hudson River Shoreline
- Recon Area 2 Iona Marsh Shoreline
- Recon Area 3 Western Shoreline Between Iona and Round Island
- Recon Area 4 CSX Railroad Tracks (East and West Sides)
- Sediment Sample Location

0 400 800
Feet
1 in = 400 ft

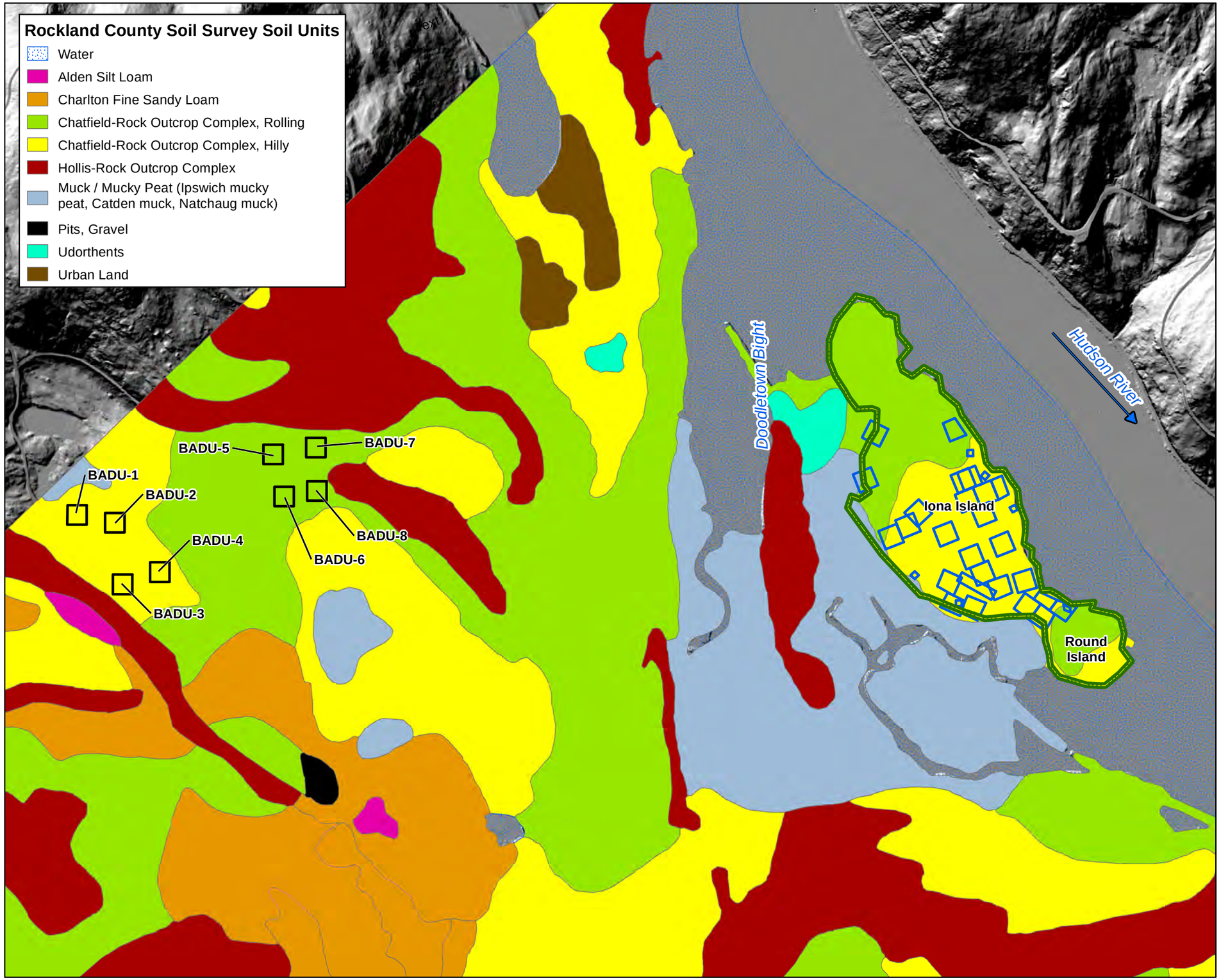
N

FIGURE 3
Phase I Shoreline Reconnaissance Survey Areas and Sediment Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Source
DEM: 2012, Photo Science, Inc., Coastal New York LiDAR (Hydro Flattened Raster DEM), 1-Meter Resolution, New York State GIS
Upland and Marsh/Wetland: Cornell Cornell Institute for Resource Information Sciences (IRIS) 2011
Map Date: 9/4/2020

\\lovetonGIS\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\HTRW\Phase\TechMemo\Figure3 Shoreline Recon Survey Areas and Sediment Sampling Locations.mxd



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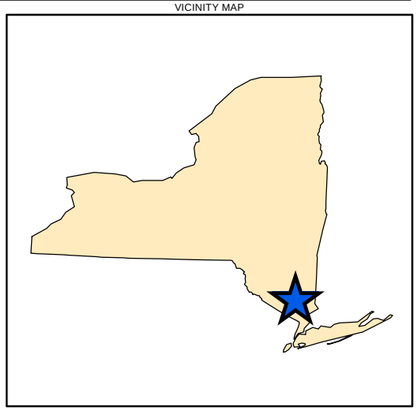

Legend

- FUDS Boundary
- Bear Mountain State Park 200x200 ft Surface and Subsurface Soil DU
- Onsite DU

0 1,000 2,000
Feet
1 in = 1,000 ft

N

FIGURE 4
Phase I Background Soil Sampling Locations

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Sources
DEM: 2012, Photo Science, Inc., Coastal New York LiDAR (Hydro Flattened Raster DEM), 1-Meter Resolution, New York State GIS
Soil Units: USDA NRCS December 2013

Map Date: 9/4/2020

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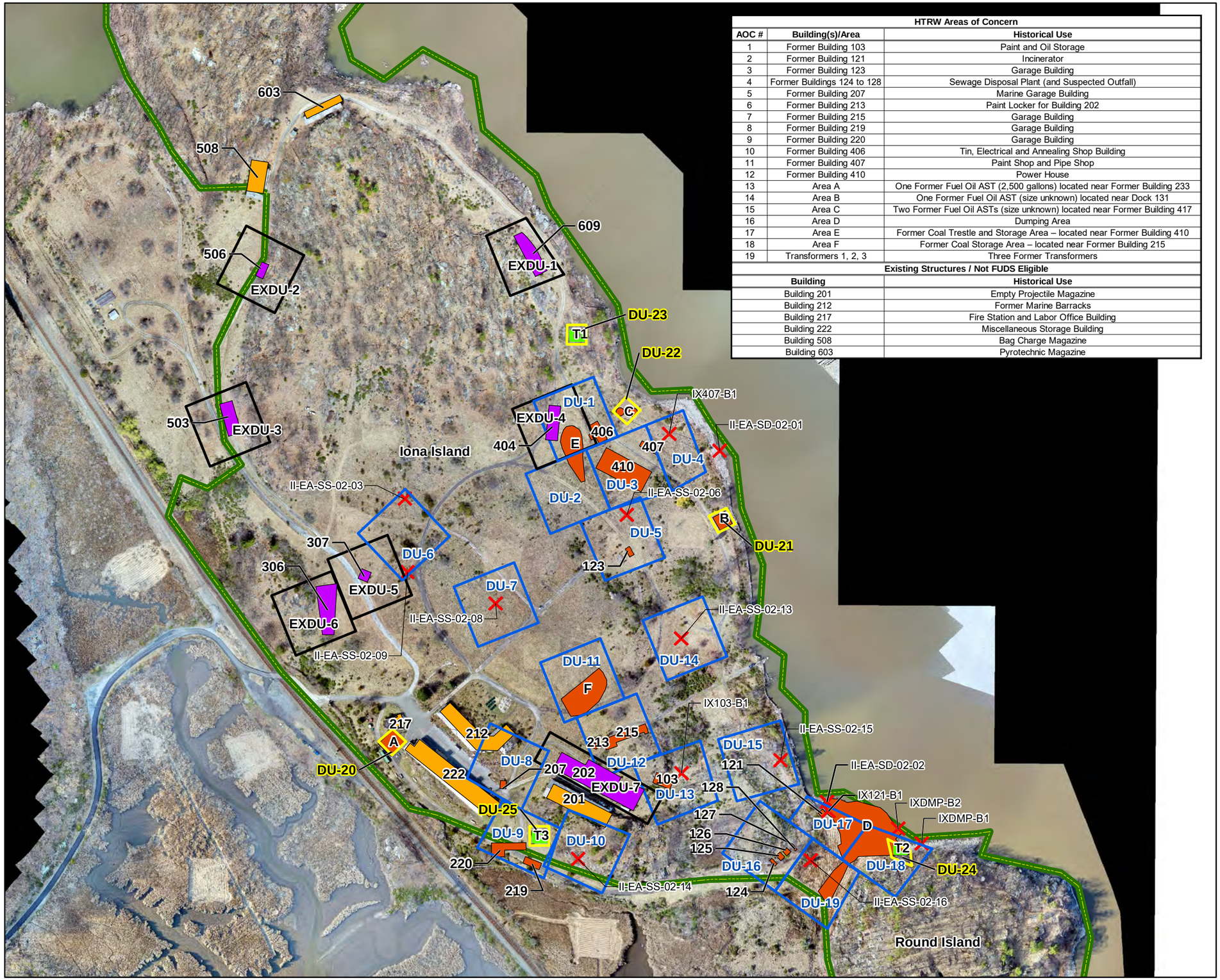
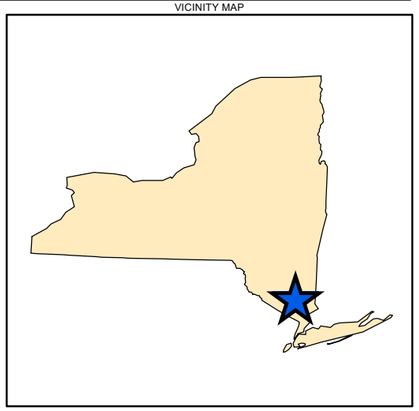


HTRW Areas of Concern		
AOC #	Building(s)/Area	Historical Use
1	Former Building 103	Paint and Oil Storage
2	Former Building 121	Incinerator
3	Former Building 123	Garage Building
4	Former Buildings 124 to 128	Sewage Disposal Plant (and Suspected Outfall)
5	Former Building 207	Marine Garage Building
6	Former Building 213	Paint Locker for Building 202
7	Former Building 215	Garage Building
8	Former Building 219	Garage Building
9	Former Building 220	Garage Building
10	Former Building 406	Tin, Electrical and Annealing Shop Building
11	Former Building 407	Paint Shop and Pipe Shop
12	Former Building 410	Power House
13	Area A	One Former Fuel Oil AST (2,500 gallons) located near Former Building 233
14	Area B	One Former Fuel Oil AST (size unknown) located near Dock 131
15	Area C	Two Former Fuel Oil ASTs (size unknown) located near Former Building 417
16	Area D	Dumping Area
17	Area E	Former Coal Trestle and Storage Area – located near Former Building 410
18	Area F	Former Coal Storage Area – located near Former Building 215
19	Transformers 1, 2, 3	Three Former Transformers
Existing Structures / Not FUDS Eligible		
Building		**Historical Use**
Building 201		Empty Projectile Magazine
Building 212		Former Marine Barracks
Building 217		Fire Station and Labor Office Building
Building 222		Miscellaneous Storage Building
Building 508		Bag Charge Magazine
Building 603		Pyrotechnic Magazine

VICINITY MAP



Legend

- FUDS Boundary
- HTRW Project Areas of Concern
- FormerTransformers
- Former Buildings with Significant Potential for Explosive Hazard (Included in the HTRW RI pending direction of contracting offer)
- Remaining Structure (Not FUDS Eligible)
- Previous Elevated Concentration of COPC
- 1-Acre DU
- 60 x 60 ft DU
- Explosives Building DU

0 300 600
Feet
1 in = 300 ft

N

FIGURE 5
HTRW Project Incremental Sampling Decision Units

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
PROJECT REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Source
Aerial: EA Engineering, Science, and Technology. April, 7 2020

Map Date: 8/21/2020

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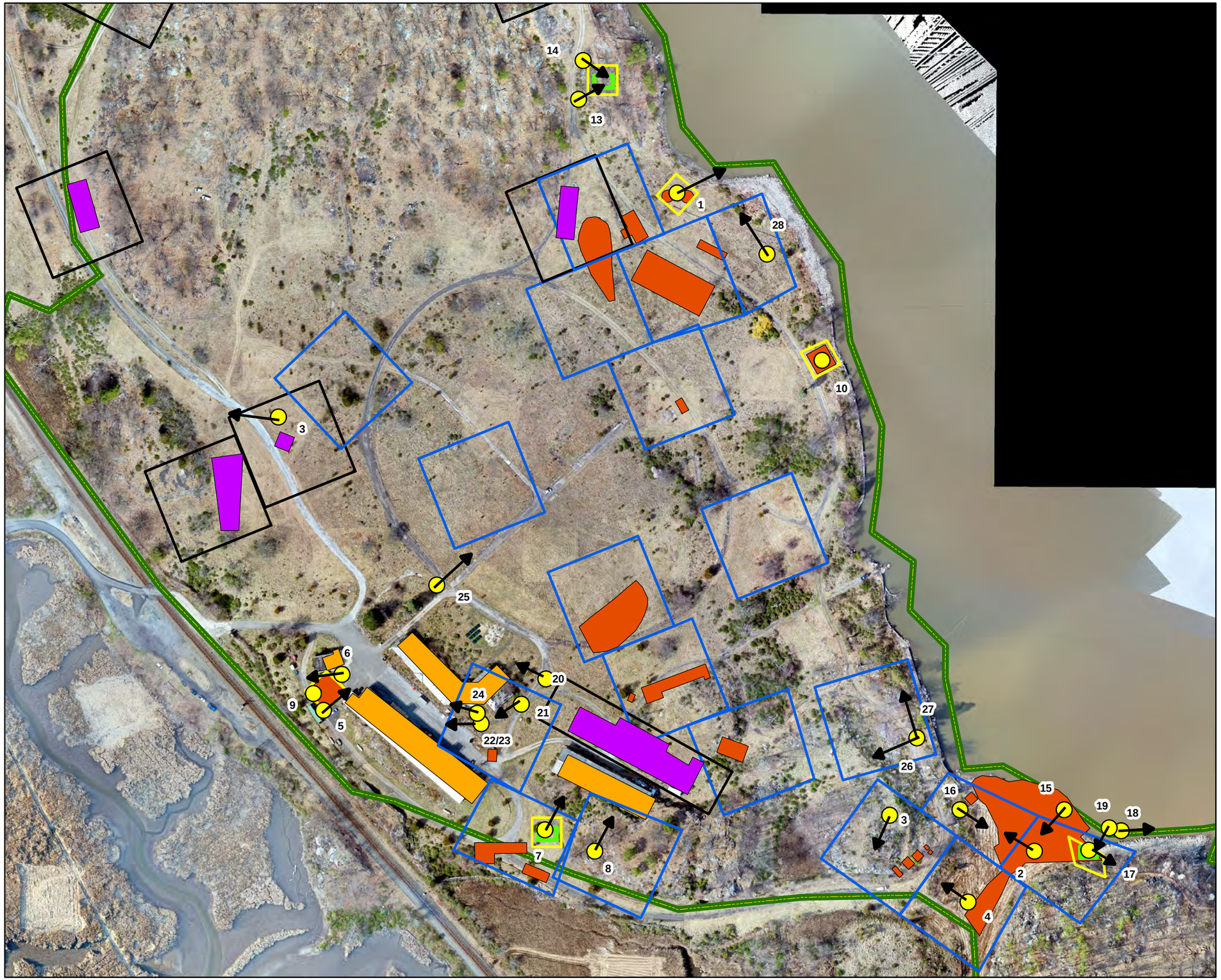
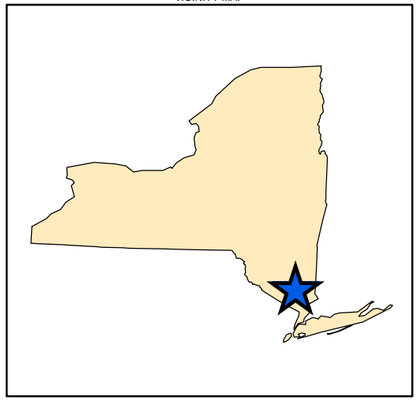


VICINITY MAP



Legend

- FUDS Boundary
- Former Buildings with Significant Potential for Explosive Hazard
- HTRW Project Areas of Concern
- Remaining Structure (Not FUDS Eligible)
- 1-Acre DU
- 60 x 60 ft DU
- Explosives Building DU
- Photo Location
- Photo Location Direction

0 200 400
Feet
1 in = 200 ft

N

FIGURE 6
Photo Location Map

HAZARDOUS TOXIC AND RADIOACTIVE WASTE
PROJECT REMEDIAL INVESTIGATION
PHASE I MEMORANDUM

Source
Aerial: EA Engineering, Science, and Technology. April 7, 2020

Map Date: 8/21/2020

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TABLES

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Table 1 Areas of Concern for Phase I Investigation

AOC Number	Building/Structure	Historical Activities	Potential AOC-Specific Contaminants	Historical Sampling Results[1,2]	Potential Release to Environment	COPC Specific Analyte List	Decision Unit
1	Former Building 103	Storage of paint and oil; likely solvent use.	Solvents from paints (VOCs and SVOCs); Petroleum Oil Lubricants (POL); and metals.	October 1996 – elevated metals (arsenic, lead) and PAHs in shallow subsurface soil sample IX103-B1 December 2007 – no samples collected	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years). SVOCs from paints would have polymerized.	TAL metals, PAHs	DU-13
2	Former Building 121	Former incinerator; historical records do not indicate whether municipal solid waste or hazardous waste was burned in the incinerator.	Residual organics and metals; PAHs from incomplete combustion.	October 1996 – elevated metals (arsenic, barium, cadmium, lead) and PAHs in shallow subsurface soil sample IX121-B1 December 2007 – elevated metals (lead) detected in sediment sample II-EA-SD-02-02 collected near this AOC	Volatilization of ash particles into air and soil deposition; potential land disposal; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater.	TAL metals, PAHs	DU-17
3	Former Building 123	Former garage; historical records do not indicate if this building was used for equipment or materials storage.	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. Note: VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-5
4	Former Buildings 124, 125, 126, 127, and 128	Sewage Disposal Plant.	Chlorides, organic compounds (VOCs and SVOCs), metals, microorganisms.	October 1996 – elevated metals (arsenic, lead) in shallow subsurface soil sample IX124/8-B1. Low concentrations of PAHs (below screening criteria). December 2007 – elevated metals (antimony, copper, lead, mercury, zinc) in surface soil sample II-EA-SS-02-16	Chemical spills; leaks from sewer lines/ tanks to surface water or groundwater; land disposal of sludge. Note: VOCs (including from POLs) are unlikely to be present in soil due to characteristics of VOCs (high solubility and mobility) and due to the time since deactivation (70 years). Due to the time since deactivation (70 years), microrganisms from DoD use are likely not present.	TAL metals, PAHs	DU-16
5	Former Building 207	Former garage used by the Marines; historical records do not indicate if this building was used for equipment or materials storage.	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-8
6	Former Building 213	Storage of paint; likely solvent use.	Solvents from paints (VOCs and SVOCs) and metals	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years). SVOCs from paints would have polymerized.	TAL metals, PAHs	DU-12
7	Former Building 215	Former garage; historical records do not indicate if this building was used for equipment or materials storage.	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-12
8	Former Building 219	Former garage; historical records do not indicate if this building was used for equipment or materials storage.	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-9
9	Former Building 220	Former garage; historical records do not indicate if this building was used for equipment or materials storage.	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-9

Table 1 Areas of Concern for Phase I Investigation

AOC Number	Building/Structure	Historical Activities	Potential AOC-Specific Contaminants	Historical Sampling Results[1,2]	Potential Release to Environment	COPC Specific Analyte List	Decision Unit
10	Former Building 406	Tin, electrical and annealing shop	Solvents (VOCs and SVOCs), POL, and metals.	No samples collected to date	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-1
11	Former Building 407	Paint shop and pipe shop; likely solvent use	Solvents (VOCs and SVOCs), POL, and metals	October 1996 – elevated metals (arsenic, lead) and PAHs in shallow subsurface soil sample IX407-B1 December 2007 – elevated metals in surface soil sample (II-EA-SS-02-05) collected near this AOC	Volatilization of VOCs to air; spills to surface soil; overland flow to surface water/ sediment; leaching to subsurface soil and groundwater. **Note:** VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL metals, PAHs	DU-3
12	Former Building 410	Power house; coal ash (including fly ash, bottom ash, and/or sludge).	PAHs and metals (such as arsenic, cadmium, and mercury)	October 1996 – highest detected PAHs at site, in shallow subsurface soil sample IX410-B1; metals were not tested December 2007 – no samples collected	Volatilization of ash particles into air and soil deposition; potential land disposal; leaching to subsurface soil and groundwater; overland flow to surface water / sediment.	TAL metals, PAHs	DU-3
13	Former fuel oil AST	One former 2,500-gallon fuel oil AST located near Former Building 233.	VOCs/BTEX, PAHs	October 1996 - no site-related VOCs in shallow subsurface soil.	Leaching to subsurface soil and groundwater; overland flow to surface water / sediment. Note: VOCs are unlikely to be present in soil due to characteristics of VOCs (high solubility and mobility) and due to the time since deactivation (70 years); however, as BTEX is a primary component of fuel oil, BTEX is retained for further evaluation.	BTEX, PAHs	DU-20
14	Former fuel oil AST	One former fuel oil AST (size unknown) located near Dock 131.	VOCs/BTEX, PAHs	No samples collected to date	Leaching to subsurface soil and groundwater; overland flow to surface water / sediment. Note: VOCs are unlikely to be present in soil due to characteristics of VOCs (high solubility and mobility) and due to the time since deactivation (70 years); however, as BTEX is a primary component of fuel oil, BTEX is retained for further evaluation.	BTEX, PAHs	DU-21
15	Former fuel oil AST	Two former fuel oil ASTs (size unknown) located near Former Building 410.	VOCs/BTEX, PAHs	October 1996 - no site-related VOCs in shallow subsurface soil.	Leaching to subsurface soil and groundwater; overland flow to surface water / sediment. Note: VOCs are unlikely to be present in soil due to characteristics of VOCs (high solubility and mobility) and due to the time since deactivation (70 years); however, as BTEX is a primary component of fuel oil, BTEX is retained for further evaluation.	BTEX, PAHs	DU-22
16	Dump Area	Historical records suggest that construction debris was used as fill between Iona and Round Island; the content of the dump is unknown but may have contained waste or munitions debris.	Unknown contents – could include solvents, paints, POL (VOCs and SVOCs); PCBs, explosives, and metals	October 1996 – elevated metals (arsenic, barium, cadmium, chromium, lead, silver); PAHs; explosives compounds (2,4-dinitrotoluene, 2,6-dinitrotoluene) in shallow subsurface soil samples IXDMP-B1, -B2; one PCB compound (Aroclor 1260) detected in sample IXDMP-B1 December 2007 – no samples collected	Leaching to subsurface soil and groundwater; overland flow to surface water / sediment. Note: VOCs (including from POLs) are unlikely to be present in soil due to characteristics of solvent related VOCs (high solubility and mobility) and due to the time since deactivation (70 years).	TAL Metals, PAHs, PCBs, Explosives	DU-17, DU-18, DU-19
17	Former Coal Trestle and Storage Area – located near Former Building 410	Coal storage for the power house.	Metals (such as arsenic, cadmium, and mercury), SVOCs	No known sampling to date	Soil deposition; leaching to subsurface soil and groundwater; soil deposition; potential land disposal.	TAL metals, PAHs	DU--1, DU-2
18	Former Coal Storage Area – located near Former Building 215	Coal stored on a concrete foundation for use at the Depot.	Metals (such as arsenic, cadmium, and mercury), SVOCs	No samples collected to date	Soil deposition; leaching to subsurface soil and groundwater; soil deposition; potential land disposal.	TAL metals, PAHs	DU-11
19	Former transformers	Electrical transformersfor Depot.	PCBs	No known sampling to date	Leaching to subsurface soil and groundwater; overland flow to surface water / sediment.	PCBs.	DU-23, DU-24, DU-25

NOTES:
1. Low (estimated) detections of 1,1-dichloroethene, acetone, and methylene chloride were detected in all of the samples analyzed for VOCs in October 1996. The samples were qualified as "B" because the compounds were detected in one or more of the QA/QC blank samples and/or "J" because the detected concentration was less than the quantitation limit.
2. The December 2007 MMRP SI samples were not selected based on proximity to the HTRW AOCs (sampled previously by Greeley), they were selected for broad coverage of the 1903 Explosion Area.

NOTES:
AOC = Area of concern
AST = Above ground storage tank
BTEX = Benzene, toluene, ethylbenzene, and xylenes
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl
POL = Petroleum oil and lubricants
SOP = Standard operating procedure
SVOC = Semi-volatile organic compound
TAL = Target analyte list
VOC = Volatile organic compound

Table 2 Former Buildings on Iona Island Naval Ammunition Depot Associated with Ammunition and Explosives

Building No., [a, b, c, f, 1]	Building Name, [a, b, c, f]	Decision Unit	Year Built [a, c]	Built Purpose or Use [c, e]	Foundation [a, b, e]	Notes [b, c, d]	Extent of Explosives Residues Presence (EM 385-1-97)			Other Basis/Notes
							Table I.5-1 Item No. [2]	Solid, Liquid, Gas	Significant or Limited	
202	Shell House #5 & Explosive "D" Plant	EXDU-7	1912	(Shop)[e]	Stone/Concrete	Walls-brick; Floor – wood; Roof – Tin laid on wood, with wood rafters ([b]) "Ammunition is almost always present" ([b]) Explosive "D" Loading Plant (g) Two buildings labeled Vacuum System Housing (no building number) are located on the right and left sides of Building 202, map dated 1947. (e)	22, 54, 63, 67-74, 97	Solid (dusts)	Significant	"D Plant" Bulk ammonium picrate? No production at Iona Depot. Possible loading of explosives into large projectiles? Document on Storage references the erection of Special buildings for "sifting" explosive "D" circa 1945. (g) This process could possibly require an explosives dust vacuum collection system. Listed on 1958 Real Estate Annex Building 226 was a one-story concrete lean-to attached to Building 202 (k).
306	Filling House #2 Cartridge Filling House and Reforming Plant (e)	EXDU-6	1939	Maintenance (Shop)[e] Maintenance (Cartridge Filling Shop) (k)	Concrete	Attached to Bldg. 313, Cartridge Case Storage([c]) This building was attached to Building 313 and had a Vertical Electric Conveyor System to assist in moving material (k).	54, 97	Solid	Significant	""At intervals, a small amount of ammunition may be kept in them for a short period but not generally" ([b]) Possible loading of explosives? Map dated 1947 shows this building was used to fill cartridge Cases(e)
307	Filling House #5 Projectile Filling House (e)	EXDU-5	1940	Maintenance (Shop)[e] Maintenance (Projectile Filling House) (k)	Concrete		54, 63, 67-74, 97	Solid	Significant	"At intervals, a small amount of ammunition may be kept in them for a short period but not generally" ([b]) Possible loading of explosives? Map dated 1947 shows this building was used to fill projectiles (e)
404	Filling House #1 Bag Charge Filling House (e)	EXDU-4	1939	Maintenance (Ammunition and Explosives) (Shop)[e]	Concrete	Attached to Bldg. 404, Powder Storage Building([c]) This building is attached to Building 421 (k)	54, 97	Solid	Significant	"At intervals, a small amount of ammunition may be kept in them for a short period but not generally" ([b]) Possible loading of explosives?
503	Filling House #6 Charge Assembly House (e)	EXDU-3	1942	Maintenance (Ammunition and Explosives) (Shop)[e]	Concrete	Powder awaiting sale ([b]) "Ammunition is almost always present" ([b])	54, 63, 67-74, 82 97	Solid	Significant	Possible assembly of cartridges, shrapnel rounds. Possible loading of explosives? Enclosure B, para 2 (i) Use of fusible links on opening between rooms normally indicates that maintenance on ammunition is being performed. This could be anything from repackaging to rebuilding.

506	Filling House #3 (Bombproof) Bombproof (e)	EXDU-2	1906	Maintenance (Storage)[f] Maintenance (Ammunition and Explosives) (k)	Brick		54, 59, 63, 67-74, 97	Solid	Significant	"At intervals, a small amount of ammunition may be kept in them for a short period but not generally" ([b]) Possible loading of explosives? Maintenance on ammunition involved a number of operations such as those listed in the 1928 Annual report. (j) Shown on map dated 1947 as only a Bombproof. (e)
609	Mine Loading Plant Inert Storehouse (f) Miscellaneous Storehouse (e)	EXDU-1	1918	Ammunition Storage (Storage)[e]	Concrete	"Ammunition is almost always present" ([b]) 8", 10", 14" and 16" dummy charges, steel plugs, 3" and 5" covers, drill projectiles, flashless pellets. (f) Mine filling operation with melted TNT. (h)	5, 48, 54, 56, 97	Solid, Liquids and Gas	Significant	Document date 17 Dec. 1942 Shown as Mine Loading Plant on map dated 1928-12-31 (e) Plant was discontinued by late 1942 and kettles used to melt TNT were removed. (h) Shown on a map dated 1947 as a Miscellaneous Storehouse (e) See 1918 Annual Report/Navy Department on detail info on Mine Loading Plant. (l)

[a]1998, ASR, Appendix E; Public Works of the Navy Building List, July 1939
[b]1998, ASR, Appendix E; Public Works Memo, 9 November 1927
[c]1998, ASR, Appendix F; Legal Description of Iona Island, 1940 (date uncertain)
[d]1998 ASR, Appendix E; 1995 Findings of Fact, Site Survey Summary Sheet, dated 30 October 1992
[e]2018 HPA, 1965 Appraisal of Iona and Round Island

(f) 1944-10-07 Ltr. Monthly Rpt. of Stowage of Ammunition (reports use of building and type of ammunition stored in 1944.
(g) 1945-00-00 Comments on building Explosive "D: Plants
(ih) 1942-12-17 Survey Request, Disposal of Kettles removed from Mine Loading Plant
(i) 1946-02-11 Report of Inspection of NAD Iona Island, NY, see Enclosure A, para 3 for burning grounds, Enclosure B, para 2 for fusible links in building 503, para. 2 and 3 for chemical stored in magazines 17 and 18.
(j) 1928-08-23 Annual Report, examples of ammunition maintenance activities
(k) 1958-05-01 Real Estate and Building Annex
(l) 1918 Annual Report/Navy Department, Mine Loading Plant

Note 2 Explanations of type of ammunition or explosives operation/facility phase of explosive released from Table I.5-1/EM 385-1-97 is provided below:

5 – Cast loading
22 –Explosives or explosives dust vacuum collection
54 – Pack/unpack bay in a maintenance or production building
56- 63 – Pressing, low speed, automated or manual (normally used in the press loading of larger items, such as projectiles and warheads
67 – Projectile base plate assembly or removal
68 – Projectile crimping
69 – Projectile fin assembly or removal
70 – Projectile main charge pressing
71 – Projectile ogive assembly or removal
72 – Projectile pull-apart (from cartridge case)
73 – Projectile rotating band or obturator assembly or removal
74 – Projectile seating
94 – Storage (e.g., earth covered magazines, aboveground magazines, covered storage pads, service magazines)
97 – Temporary storage bay in a maintenance or production building

Note 1 - Building requires more information about specific operations that would indicate a source of potential significant explosives residue and would need to be revised to include operations such as cast loading (Item #5 of Table I.5-1/EM 385-1-97).

General Notes: Section 5 (*Building and Installed Equipment Containing Explosive Residues That Present Explosion Hazards*) in Engineering Manual 385-1-97 *Explosives Safety and Health Requirements Manual* and Department of Army Pamphlet 385-64 *Ammunition and Explosives Safety Standards* is specific to real property which includes buildings and installed equipment. The guidance provides safety requirements for former operating buildings and any installed equipment when there is a potential of residual explosives that present an explosive hazard.
Significant explosives residues – Caused by operations that can result in extensive migration of significant amounts of explosives in the building and its installed equipment. Can be released in solid, liquid, or vapor form. Can migrate into cracks, voids, behind wall and roof panels, drains, roof rafters, porous surfaces, etc. Solids - operations capable of generating explosive dusts result in significant migration and residual presence. Liquids - In process liquid explosives can leave residues in pipes and vessels. Spills and wash-down of equipment can carry explosives into cracks in the floor and into drains and sumps. Gases - When solid explosives are heated, can volatilize into the air, and migrate about the building. After condensation, the explosive is again a solid.

Table 3 HTRW RI Phase I Sample Collection

Decision Unit #	Sample Type	Sample Identification Code	Analyses	Sample Date	Sample Time	QA/QC Sample	COC ID	Soil pH
Phase I Investigation Area: Surface and Subsurface Soil, Groundwater								
DU-1	Surface Soil Incremental Sample collected in triplicate	IINY-DU-1A-SSIS-20200318	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/18/2020	1500	MS/MSD	031820-B	5.6
		IINY-DU-1B-SSIS-20200318		3/18/2020	1500		031820-B	6.5
		IINY-DU-1C-SSIS-20200318		3/18/2020	1500		031820-B	6.8
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-1A-SBIS1-YYYYMMDD		Advanced 6 hand auger borings at DU-1. Soil depth to refusal ranged from 6 to 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-1B-SBIS1-YYYYMMDD						
		IINY-DU-1C-SBIS1-YYYYMMDD						
DU-2	Surface Soil Incremental Sample collected in triplicate	IINY-DU-2A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/19/2020	815		031920-A	5.6
		IINY-DU-2B-SSIS-20200319		3/19/2020	815		031920-A	6.6
		IINY-DU-2C-SSIS-20200319		3/19/2020	815		031920-A	6.8
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-2A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-2. Hard to locate areas to advance borings due to exposed bedrock and shallow bedrock/refusal. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-2B-SBIS1-YYYYMMDD						
		IINY-DU-2C-SBIS1-YYYYMMDD						
DU-3	Surface Soil Incremental Sample collected in triplicate	IINY-DU-3A-SSIS-20200318	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/18/2020	1545		031820-B	7.0
		IINY-DU-3B-SSIS-20200318		3/18/2020	1545		031820-B	7.0
		IINY-DU-3C-SSIS-20200318		3/18/2020	1545		031820-B	7.1
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-3A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-3. Hard to locate areas to advance borings due to exposed bedrock and shallow bedrock/refusal. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-3B-SBIS1-YYYYMMDD						
		IINY-DU-3C-SBIS1-YYYYMMDD						
DU-4	Surface Soil Incremental Sample collected in triplicate	IINY-DU-4A-SSIS-20200318	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/18/2020	1430		031820-B	6.9
		IINY-DU-4B-SSIS-20200318		3/18/2020	1430		031820-B	7.0
		IINY-DU-4C-SSIS-20200318		3/18/2020	1430		031820-B	7.1
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-4A-SBIS1-YYYYMMDD		Advanced 6 hand auger borings at DU-4. Significant anomalies in 1-acre DU. Soil depth to refusal at one boring was 12 inches bgs, soil depth to refusal at remaining borings ranged from 4 to 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-4B-SBIS1-YYYYMMDD						
		IINY-DU-4C-SBIS1-YYYYMMDD						
DU-5	Surface Soil Incremental Sample collected in triplicate	IINY-DU-5A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/19/2020	900		031920-A	7.1
		IINY-DU-5B-SSIS-20200319		3/19/2020	900		031920-A	7.1
		IINY-DU-5C-SSIS-20200319		3/19/2020	900		031920-A	7.1
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-5A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-5. Hard to locate areas to advance borings due to exposed bedrock and shallow bedrock/refusal. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-5B-SBIS1-YYYYMMDD						
		IINY-DU-5C-SBIS1-YYYYMMDD						
DU-6	Surface Soil Incremental Sample collected in triplicate	IINY-DU-6A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, pH (screened in the field with pH meter)	3/19/2020	1500		031920-B	7.5
		IINY-DU-6B-SSIS-20200319		3/19/2020	1500		031920-B	7.5
		IINY-DU-6C-SSIS-20200319		3/19/2020	1500		031920-B	7.5
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-6A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-6. Exposed bedrock / bedrock wall in DU-6. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-6B-SBIS1-YYYYMMDD						
		IINY-DU-6C-SBIS1-YYYYMMDD						
DU-7	Surface Soil Incremental Sample collected in triplicate	IINY-DU-7A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, pH (screened in the field with pH meter)	3/19/2020	1345		031920-B	7.4
		IINY-DU-7B-SSIS-20200319		3/19/2020	1345		031920-B	7.4
		IINY-DU-7C-SSIS-20200319		3/19/2020	1345		031920-B	7.5
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-7A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-7. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-7B-SBIS1-YYYYMMDD						
		IINY-DU-7C-SBIS1-YYYYMMDD						
DU-8	Surface Soil Incremental Sample collected in triplicate	IINY-DU-8A-SSIS-YYYYMMDD	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	DU-8 Deletion – DU-8 was placed at former Building 207 that was a small garage located between existing Buildings 222, 201, and 212. Building 207 is also known as HTRW AOC #5. DU-8 is a 200x200 ft decision unit that was established around former Building 207 to determine whether former operations at this building served as a source of contaminants to the environment at concentrations that may result in potential risks to human and ecological receptors. Following mark-out DU-8 in the field and site reconnaissance conducted on 18 March 2020, numerous rusted and deteriorating 55-gallon drums, deteriorating pallets, piles of roofing shingles, chain link fencing, and other debris were observed in DU-8. Several empty 55-gallon drums were lying on their sides, while other drums were upright and filled with fluid. The material contained within the drums is not known. Residue indicative of roofing tar was observed on the outside of several drums, and staining was noted on the ground surface. Per discussion with USACE on 24 March 2020, DU-8 will not be sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.				
		IINY-DU-8B-SSIS-YYYYMMDD						
		IINY-DU-8C-SSIS-YYYYMMDD						
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-8A-SBIS1-YYYYMMDD						
		IINY-DU-8B-SBIS1-YYYYMMDD						
		IINY-DU-8C-SBIS1-YYYYMMDD						
DU-9	Surface Soil Incremental Sample collected in triplicate	IINY-DU-9A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/19/2020	1040		031920-A	7.1
		IINY-DU-9B-SSIS-20200319		3/19/2020	1040		031920-B	6.9
		IINY-DU-9C-SSIS-20200319		3/19/2020	1040	MS/MSD	031920-B	7.0
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-9A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-9. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-9B-SBIS1-YYYYMMDD						
		IINY-DU-9C-SBIS1-YYYYMMDD						
DU-10	Surface Soil Incremental Sample collected in triplicate	IINY-DU-10A-SSIS-20200319	TAL Metals by EPA Methods 6010C/6020A, pH (screened in the field with pH meter)	3/19/2020	1120		031920-B	7.1
		IINY-DU-10B-SSIS-20200319		3/19/2020	1120		031920-B	7.1
		IINY-DU-10C-SSIS-20200319		3/19/2020	1120		031920-B	7.2
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-10A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-10. Exposed bedrock and bedrock wall within DU. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-10B-SBIS1-YYYYMMDD						
		IINY-DU-10C-SBIS1-YYYYMMDD						
DU-11	Surface Soil Incremental Sample collected in triplicate	IINY-DU-11A-SSIS-20200330	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/30/2020	1115	MS/MSD	033020-A	7.0
		IINY-DU-11B-SSIS-20200330		3/30/2020	1115		033020-A	7.0
		IINY-DU-11C-SSIS-20200330		3/30/2020	1115		033020-A	7.2
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-11A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-11. Soil depth to refusal less than 2 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-11B-SBIS1-YYYYMMDD						
		IINY-DU-11C-SBIS1-YYYYMMDD						
DU-12	Surface Soil Incremental Sample collected in triplicate	IINY-DU-12A-SSIS-20200330	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/30/2020	1215		033020-A	5.5
		IINY-DU-12B-SSIS-20200330		3/30/2020	1215		033020-A	6.1
		IINY-DU-12C-SSIS-20200330		3/30/2020	1215		033020-A	6.3
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-12A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-12. Soil depth to refusal less than 2 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-12B-SBIS1-YYYYMMDD						
		IINY-DU-12C-SBIS1-YYYYMMDD						

Decision Unit #	Sample Type	Sample Identification Code	Analyses	Sample Date	Sample Time	QA/QC Sample	COC ID	Soil pH
Phase I Investigation Area: Surface and Subsurface Soil, Groundwater								
DU-13	Surface Soil Incremental Sample collected in triplicate	IINY-DU-13A-SSIS-20200330	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/30/2020	1340		033020-A	6.8
		IINY-DU-13B-SSIS-20200330		3/30/2020	1340		033020-A	7.0
		IINY-DU-13C-SSIS-20200330		3/30/2020	1340		033020-A	6.9
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-13A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-13. Soil depth to refusal less than 2 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-13B-SBIS1-YYYYMMDD						
		IINY-DU-13C-SBIS1-YYYYMMDD						
DU-14	Surface Soil Incremental Sample collected in triplicate	IINY-DU-14A-SSIS-20200320	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, pH (screened in the field with pH meter)	3/20/2020	830		032020-A	6.7
		IINY-DU-14B-SSIS-20200320		3/20/2020	830		302020-A	6.9
		IINY-DU-14C-SSIS-20200320		3/20/2020	830		032020-A	7.0
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-14A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-9. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-14B-SBIS1-YYYYMMDD						
		IINY-DU-14C-SBIS1-YYYYMMDD						
DU-15	Surface Soil Incremental Sample collected in triplicate	IINY-DU-15A-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, pH (screened in the field with pH meter)	4/1/2020	820		040120-A	6.2
		IINY-DU-15B-SSIS-20200401		4/1/2020	820		040120-A	6.3
		IINY-DU-15C-SSIS-20200401		4/1/2020	820		040120-A	6.6
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-15A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-15. Soil depth to refusal less than 12 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-15B-SBIS1-YYYYMMDD						
		IINY-DU-15C-SBIS1-YYYYMMDD						
DU-16	Surface Soil Incremental Sample collected in triplicate	IINY-DU-16A-SSIS-20200331	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, pH (screened in the field with pH meter)	3/31/2020	950		033120-B	6.9
		IINY-DU-16B-SSIS-20200331		3/31/2020	950		033120-B	6.9
		IINY-DU-16C-SSIS-20200331		3/31/2020	950		033120-B	7.4
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-16A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-16. Soil depth to refusal less than 12 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-16B-SBIS1-YYYYMMDD						
		IINY-DU-16C-SBIS1-YYYYMMDD						
DU-17	Surface Soil Incremental Sample collected in triplicate	IINY-DU-17A-SSIS-20200331	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, Aroclor PCBs by EPA Method 8082A, explosives by EPA Method 8330B, and pH (screened in the field with pH meter)	3/31/2020	815		033120-B	7.2
		IINY-DU-17B-SSIS-20200331		3/31/2020	815		033120-B	7.2
		IINY-DU-17C-SSIS-20200331		3/31/2020	815		033120-B	7.1
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-17A-SBIS1-20200331		3/31/2020	845		033120-A	7.0
		IINY-DU-17B-SBIS1-20200331		3/31/2020	845		033120-A	7.4
		IINY-DU-17C-SBIS1-20200331		3/31/2020	845		033120-A	7.7
DU-18	Surface Soil Incremental Sample collected in triplicate	IINY-DU-18A-SSIS-20200331	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, Aroclor PCBs by EPA Method 8082A, explosives by EPA Method 8330B, and pH (screened in the field with pH meter)	3/31/2020	1345		033120-B	7.5
		IINY-DU-18B-SSIS-20200331		3/31/2020	1345		033120-B	8.0
		IINY-DU-18C-SSIS-20200331		3/31/2020	1345		033120-B	7.8
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-18A-SBIS1-20200331		3/31/2020	1420		033120-B	8.1
		IINY-DU-18B-SBIS1-20200331		3/31/2020	1420		033120-B	8.1
		IINY-DU-18C-SBIS1-20200331		3/31/2020	1420		033120-b	8.0
DU-19	Surface Soil Incremental Sample collected in triplicate	IINY-DU-19A-SSIS-20200331	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM, Aroclor PCBs by EPA Method 8082A, explosives by EPA Method 8330B, and pH (screened in the field with pH meter)	3/31/2020	1045	MS/MSD	033120-A	7.7
		IINY-DU-19B-SSIS-20200331		3/31/2020	1045		033120-A	7.5
		IINY-DU-19C-SSIS-20200331		3/31/2020	1045		033120-A	7.4
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-19A-SBIS1-20200331		3/31/2020	1100		033120-A	7.0
		IINY-DU-19B-SBIS1-20200331		3/31/2020	1100		033120-A	7.4
		IINY-DU-19C-SBIS1-20200331		3/31/2020	1100		033120-A	7.7
DU-20	Surface Soil Incremental Sample collected in triplicate	IINY-DU-20A-SSIS-20200316	PAHs by EPA Method 8270 SIM (surface and subsurface soil), BTEX by EPA Method 8260B (subsurface soil only), and pH (screened in the field with pH meter)	3/16/2020	1530		031620-A	6.1
		IINY-DU-20B-SSIS-20200316		3/16/2020	1530		031620-A	6.5
		IINY-DU-20C-SSIS-20200316		3/16/2020	1530		031620-A	6.6
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-20A-SBIS1-20200317		3/17/2020	930	MS/MSD	031720-A / 031720-C	6.8
		IINY-DU-20B-SBIS1-20200317		3/17/2020	930		031720-A / 031720-C	6.9
		IINY-DU-20C-SBIS1-20200317		3/17/2020	930		031720-A / 031720-C	6.8
DU-21	Surface Soil Incremental Sample collected in triplicate	IINY-DU-21A-SSIS-20200317	PAHs by EPA Method 8270 SIM (surface and subsurface soil), BTEX by EPA Method 8260B (subsurface soil only), and pH (screened in the field with pH meter)	3/17/2020	1240		031720-A	6.3
		IINY-DU-21B-SSIS-20200317		3/17/2020	1240		031720-A	6.8
		IINY-DU-21C-SSIS-20200317		3/17/2020	1240		031720-A	6.8
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-21A-SBIS1-20200317		3/17/2020	1145		031720-A / 031720-C	7.0
		IINY-DU-21B-SBIS1-20200317		3/17/2020	1145		031720-A / 031720-C	7.1
		IINY-DU-21C-SBIS1-20200317		3/17/2020	1145		031720-A / 031720-C	7.3
DU-22	Surface Soil Incremental Sample collected in triplicate	IINY-DU-22A-SSIS-20200316	PAHs by EPA Method 8270 SIM (surface and subsurface soil), BTEX by EPA Method 8260B (subsurface soil only), and pH (screened in the field with pH meter)	3/16/2020	1530		031620-A	6.7
		IINY-DU-22B-SSIS-20200316		3/16/2020	1530		031620-A	6.8
		IINY-DU-22C-SSIS-20200316		3/16/2020	1530		031620-A	6.8
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-22A-SBIS1-YYYYMMDD		Advanced 6 hand auger borings at DU-22. Soil depth to refusal ranged from 6 to 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-22B-SBIS1-YYYYMMDD						
		IINY-DU-22C-SBIS1-YYYYMMDD						
DU-23	Surface Soil Incremental Sample collected in triplicate	IINY-DU-23A-SSIS-20200320	Aroclor PCBs by EPA Method 8082A, and pH (screened in the field with pH meter)	3/20/2020	915		032020-A	6.0
		IINY-DU-23B-SSIS-20200320		3/20/2020	915		032020-A	5.4
		IINY-DU-23C-SSIS-20200320		3/20/2020	915		032020-A	5.5
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-23A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-23. Soil depth to refusal less than 6 inches bgs (<1 ft). Majority of DU is exposed bedrock. Very shallow soil. Will not collect subsurface soil.				
		IINY-DU-23B-SBIS1-YYYYMMDD						
		IINY-DU-23C-SBIS1-YYYYMMDD						
DU-24	Surface Soil Incremental Sample collected in triplicate	IINY-DU-24A-SSIS-YYYYMMDD	Aroclor PCBs by EPA Method 8082A, and pH (screened in the field with pH meter)	DU-24 Deletion – Historical documents indicate that a former transformers on a pole was located along the former eastern sentry path from Iona Island onto Round Island. A 60x60 ft decision unit, DU-24, was established on Round Island to evaluate the presence/absence of PCBs associated with the former transformer. DU-24 was one of three transformer decision units that is known as HTRW AOC #19 on Iona Island (Note: the other two transformer decision units were collect). DU-24 is bounded by the Hudson River to east/northeast and the former dump area between Iona and Round Islands to the northwest. A downed former electric pole was noted during a reconnaissance on 20 March 2020. The majority of DU-24 is exposed bedrock with little to thin soil cover. Sediment and soil was noted along the northeastern edge of DU-24 off the bedrock outcrop of Round Island, however this sediment/soil is along the shoreline of the tidally influenced Hudson River. Per a call with USACE on 24 March 2020, DU-24 will not be sampled. There is little/no soil available for sampling in the area of the former transformer. Any PCBs that may have been released from former transformers would have been transported via runoff and overland flow across the bedrock outcrop of Round Island to the Hudson River. However, as the Hudson River itself is a PCB source, collecting PCBs from soil/sediment along the Hudson River will not provide information that could be used to evaluate a potential release related to former transformers.				
		IINY-DU-24B-SSIS-YYYYMMDD						
		IINY-DU-24C-SSIS-YYYYMMDD						
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-24A-SBIS1-YYYYMMDD						
		IINY-DU-24B-SBIS1-YYYYMMDD						
		IINY-DU-24C-SBIS1-YYYYMMDD						
DU-25	Surface Soil Incremental Sample collected in triplicate	IINY-DU-25A-SSIS-20200320	Aroclor PCBs by EPA Method 8082A, and pH (screened in the field with pH meter)	3/20/2020	1030	MS/MSD	032020-A	6.2
		IINY-DU-25B-SSIS-20200320		3/20/2020	1030	032020-A	032020-A	6.4
		IINY-DU-25C-SSIS-20200320		3/20/2020	1030		032020-A	6.4
	Subsurface Soil Incremental Sample collected in triplicate	IINY-DU-25A-SBIS1-YYYYMMDD		Advanced5 hand auger borings at DU-25. Exposed bedrock and bedrock wall within DU. Soil depth to refusal less than 8 inches bgs (<1 ft). Will not collect subsurface soil.				
		IINY-DU-25B-SBIS1-YYYYMMDD						
		IINY-DU-25C-SBIS1-YYYYMMDD						

Decision Unit #	Sample Type	Sample Identification Code	Analyses	Sample Date	Sample Time	QA/QC Sample	COC ID	Soil pH
Phase I Investigation Area: Surface and Subsurface Soil, Groundwater								
Former Buildings Identified as Having Significant Potential for Explosive Hazard (EXDU; pending direction of contracting offer): Surface and Subsurface Soil								
EXDU-1	Surface Soil Incremental Sample collected in triplicate	IINY-DU-1A-SSIS-20200317	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/17/2020	1535		031720-B	7.0
		IINY-DU-1B-SSIS-20200317		3/17/2020	1535		031720-B	6.9
		IINY-DU-1C-SSIS-20200317		3/17/2020	1535		031720-B	6.9
EXDU-2	Surface Soil Incremental Sample collected in triplicate	IINY-DU-2A-SSIS-20200317	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/17/2020	1610		031720-B	7.0
		IINY-DU-2B-SSIS-20200317		3/17/2020	1610		031720-B	6.8
		IINY-DU-2C-SSIS-20200317		3/17/2020	1610		031720-B	6.9
EXDU-3	Surface Soil Incremental Sample collected in triplicate	IINY-DU-3A-SSIS-20200318	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/18/2020	800		031820-A	6.3
		IINY-DU-3B-SSIS-20200318		3/18/2020	800		031820-A	6.5
		IINY-DU-3C-SSIS-20200318		3/18/2020	800		031820-A	5.5
EXDU-4	Surface Soil Incremental Sample collected in triplicate	IINY-DU-4A-SSIS-20200317	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/17/2020	1430	MS/MSD	031720-A	6.5
		IINY-DU-4B-SSIS-20200317		3/17/2020	1430		031720-B	6.3
		IINY-DU-4C-SSIS-20200317		3/17/2020	1430		031720-B	6.8
EXDU-5	Surface Soil Incremental Sample collected in triplicate	IINY-DU-5A-SSIS-20200318	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/18/2020	930		031820-A	7.1
		IINY-DU-5B-SSIS-20200318		3/18/2020	930		031820-A	7.2
		IINY-DU-5C-SSIS-20200318		3/18/2020	930		031820-A	7.3
EXDU-6	Surface Soil Incremental Sample collected in triplicate	IINY-DU-6A-SSIS-20200318	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/18/2020	1015		031820-A	7.2
		IINY-DU-6B-SSIS-20200318		3/18/2020	1015		031820-A	7.3
		IINY-DU-6C-SSIS-20200318		3/18/2020	1015		031820-A	7.3
EXDU-7	Surface Soil Incremental Sample collected in triplicate	IINY-DU-7A-SSIS-20200318	Explosives residues by EPA Method 8330B, and pH (screened in the field with pH meter)	3/18/2020	1145	MS/MSD	031820-A	7.0
		IINY-DU-7B-SSIS-20200318		3/18/2020	1145		031820-A	7.0
		IINY-DU-7C-SSIS-20200318		3/18/2020	1145		031820-A	7.1
Phase I Background: Surface and Subsurface Soil								
BDU-1	Surface Soil Incremental Sample	IINY-BADU-1-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	1145		040120-A	6.3
	Subsurface Soil Incremental Sample	IINY-BADU-1-SBIS1-YYYYMMDD		<12" to refusal. Did not collect subsurface soil.				
BDU-2	Surface Soil Incremental Sample	IINY-BADU-2-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	1215		040120-A	6.7
	Subsurface Soil Incremental Sample	IINY-BADU-2-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-3	Surface Soil Incremental Sample	IINY-BADU-3-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	1245		040120-A	6.0
	Subsurface Soil Incremental Sample	IINY-BADU-3-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-4	Surface Soil Incremental Sample	IINY-BADU-4-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	1320		040120-A	6.1
	Subsurface Soil Incremental Sample	IINY-BADU-4-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-5	Surface Soil Incremental Sample	IINY-BADU-5-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	955	MS/MSD	040120-A	6.1
	Subsurface Soil Incremental Sample	IINY-BADU-5-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-6	Surface Soil Incremental Sample	IINY-BADU-6-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	920		040120-A	5.9
	Subsurface Soil Incremental Sample	IINY-BADU-6-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-7	Surface Soil Incremental Sample	IINY-BADU-7-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	900		040120-A	6.1
	Subsurface Soil Incremental Sample	IINY-BADU-7-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
BDU-8	Surface Soil Incremental Sample	IINY-BADU-8-SSIS-20200401	TAL Metals by EPA Methods 6010C/6020A, Mercury by EPA Method 7471B, PAHs by EPA Method 8270 SIM	4/1/2020	925		040120-A	6.0
	Subsurface Soil Incremental Sample	IINY-BADU-8-SBIS1-YYYYMMDD		<12" to refusal. Portions of DU is rock outcrop. Soil typically only 3" to 8" in depth. Did not collect subsurface soil.				
QA/QC samples								
TB	Grab	TB03172020	VOCs by 8260B	3/17/2020	1500	Trip Blank (VOCs)	031720-C	N/A
FB	Grab	FB03172020	VOCs by 8260B	3/17/2020	1545	Field Blank (VOCs)	031720-C	N/A

Note:
BADU = Background area decision unit
COC = Chain of custody
DU = Decision Unit
EPA = United States Environmental Protection Agency
EXDU = Explosives building decision unit
FB = Field blank
ID = Identification
MS = Matrix spike
MSD = Matrix spike duplicate
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl
QA = Quality assurance
QC = Quality control
SIM = Selected ion monitoring
TAL = Target analyte list
TB = Trip blank
VOC = Volatile organic compound

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Table 4a DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit	DU-1 (AOC #17: Area E – Former Coal Trestle and Storage Area [Located Near Former Building 410] and AOC #10: Former Building 406 – Tin, Electrical, and Annealing Building Shop)						% RSD	95% Upper Confidence Limit Chebychev Test
Sample ID					IINY-DU-1A-SSIS-20200318		IINY-DU-1B-SSIS-20200318		IINY-DU-1C-SSIS-20200318			
Depth (in.)					0-6		0-6		0-6			
Matrix					Surface Soil		Surface Soil		Surface Soil			
Sample Date					3/18/2020 15:00		3/18/2020 15:00		3/18/2020 15:00			
Soil pH												
pH	N/A	N/A	N/A	N/A	5.6		6.5		6.8		9.91	7.87
Mercury												
Mercury	7439-97-6	1.1	4.6	mg/kg	0.084		0.08		0.078		4	0.0884
Target Analyte List Metals												
Aluminum	7429-90-5	7,700	110,000	mg/kg	11,000		11,000		11,000		0	11,000
Antimony	7440-36-0	3.1	47	mg/kg	0.52		0.51		0.47		5.29	0.567
Arsenic	7440-38-2	0.68	3	mg/kg	11		12		8.9	J	14.9	14.6
Barium	7440-39-3	1,500	22,000	mg/kg	71		66		65		4.77	75.4
Beryllium	7440-41-7	16	230	mg/kg	0.52		0.5		0.55		4.81	0.587
Cadmium	7440-43-9	7.1	98	mg/kg	0.35		0.26		0.24		20.7	0.431
Calcium	7440-70-2	NS	NS	mg/kg	3,700		5,100		4,800		16.3	6,390
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	14		13		13		4.33	14.8
Cobalt	7440-48-4	2.3	35	mg/kg	7		6.8		7.5		5.08	8.01
Copper	7440-50-8	310	4,700	mg/kg	21		22		21		2.71	22.8
Iron	7439-89-6	5,500	82,000	mg/kg	16,000		16,000		15,000		3.69	17,100
Lead	7439-92-1	400	800	mg/kg	84		57		52		26.8	108
Magnesium	7439-95-4	NS	NS	mg/kg	3800		4700		4300		10.6	5400
Manganese	7439-96-5	180	2,600	mg/kg	470		470		480		1.22	488
Nickel	7440-02-0	150	2,200	mg/kg	14		14		14		0	14
Potassium	2023695	NS	NS	mg/kg	1,200		1,200		1,200		0	1,200
Selenium	7782-49-2	39	580	mg/kg	0.44	J	0.32	J	0.31	J	20.3	0.539
Silver	7440-22-4	39	580	mg/kg	0.093	U (LOQ)	0.099	U (LOQ)	0.099	U (LOQ)	ND	ND
Sodium	7440-23-5	NS	NS	mg/kg	95	J	100	J	110	J	7.51	121
Thallium	7440-28-0	0.078	1.2	mg/kg	0.14		0.12		0.12		9.12	0.156
Vanadium	7440-62-2	39	580	mg/kg	23		21		22		4.55	24.5
Zinc	7440-66-6	2,300	35,000	mg/kg	92	J	76	J	67	J	16.2	110
Polycyclic Aromatic Hydrocarbons												
1-Methylnaphthalene	90-12-0	18	73	mg/kg	**0.0058**	J	**0.0044**	J	**0.0036**	J	24.2	0.0074
2-Methylnaphthalene	91-57-6	24	300	mg/kg	**0.0098**	J	**0.0062**	J	**0.0053**	J	33.500	0.0131
Acenaphthene	83-32-9	360	4,500	mg/kg	**0.007**	J	**0.0068**	J	**0.0054**	J	13.600	0.00859
Acenaphthylene	208-96-8	NS	NS	mg/kg	**0.041**	J	**0.027**	J	**0.026**	J	26.800	0.0524
Anthracene	120-12-7	1,800	23,000	mg/kg	**0.032**	J	**0.028**	J	**0.025**	J	12.400	0.0372
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	**0.15**	J	**0.13**	J	**0.11**	J	15.400	0.18
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	**0.17**	J	**0.15**	J	**0.13**	J	13.300	0.2
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	**0.27**	J	**0.23**	J	**0.2**	J	15.100	0.322
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	**0.13**	J	**0.12**	J	**0.1**	J	13.100	0.155
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	**0.092**	J	**0.076**	J	**0.065**	J	17.500	0.112
Chrysene	218-01-9	110	2,100	mg/kg	**0.17**	J	**0.14**	J	**0.12**	J	17.600	0.207
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	**0.035**	J	**0.03**	J	**0.026**	J	14.900	0.0417
Fluoranthene	206-44-0	240	3,000	mg/kg	**0.26**	J	**0.24**	J	**0.2**	J	13.100	0.31
Fluorene	86-73-7	240	3,000	mg/kg	**0.009**	J	**0.0077**	J	**0.008**	J	8.270	0.00995
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	**0.15**	J	**0.14**	J	**0.12**	J	11.200	0.175
Naphthalene	91-20-3	2	8.6	mg/kg	**0.011**	J	**0.0075**	J	**0.0063**	J	29.500	0.0144
Phenanthrene	85-01-8	NS	NS	mg/kg	**0.084**	J	**0.09**	J	**0.074**	J	9.780	0.103
Pyrene	129-00-0	180	2,300	mg/kg	**0.26**	J	**0.24**	J	**0.2**	J	13.100	0.31
Total LMW PAHs	N/A	NS	NS	mg/kg	**0.3496**		**0.3076**		**0.2636**		14	0.415
Total HMW PAHs	N/A	NS	NS	mg/kg	**1.7766**		**1.5636**		**1.3346**		14.2	2.11

Analyte	DU-2 (AOC #17: Area E – Former Coal Trestle and Storage Area [Located Near Former Building 410])						% RSD	95% Upper Confidence Limit Chebychev Test
Sample ID	IINY-DU-2A-SSIS-20200319		IINY-DU-2B-SSIS-20200319		IINY-DU-2C-SSIS-20200319			
Depth (in.)	0-6		0-6		0-6			
Matrix	Surface Soil		Surface Soil		Surface Soil			
Sample Date	3/19/2020 8:15		3/19/2020 8:15		3/19/2020 8:15			
Soil pH								
pH	5.6		6.6		6.8		10.2	7.95
Mercury								
Mercury	0.091		0.092		0.11		11	0.125
Target Analyte List Metals								
Aluminum	13,000		13,000		13,000		0	13,000
Antimony	0.61		0.62		0.68		5.95	0.732
Arsenic	14		13		14		4.22	15.1
Barium	68		74		79		7.48	87.5
Beryllium	0.54		0.55		0.65		10.5	0.733
Cadmium	0.2		0.23		0.25		11.1	0.29
Calcium	13,000		11,000		7,800		24.7	17,200
Chromium, Total	15		15		15		0	15
Cobalt	7.6		7.4		7.7		2.02	7.95
Copper	27		26		28		3.7	29.5
Iron	19,000		18,000		19,000		3.09	20,100
Lead	78		93		110		17.1	134
Magnesium	10,000		9,000		7,300		15.6	12,200
Manganese	620		660		640		3.12	690
Nickel	17		16		18		5.88	19.5
Potassium	1,400		1,300		1,200		7.69	1,550
Selenium	0.31	J	0.33	J	0.32	J	3.13	0.345
Silver	0.097	U (LOQ)	0.097	U (LOQ)	0.1	U (LOQ)	ND	ND
Sodium	75	J	75	J	71	J	3.13	79.5
Thallium	0.13		0.13		0.13		0	0.13
Vanadium	21		21		23		5.33	24.6
Zinc	72		74		80		5.53	85.8
Polycyclic Aromatic Hydrocarbons								
1-Methylnaphthalene	0.075		0.036		0.046		38.7	0.103
2-Methylnaphthalene	0.055		0.031		0.04		28.9	0.0725
Acenaphthene	0.26		0.12		0.15		41.7	0.362
Acenaphthylene	0.14		0.1		0.09		24.1	0.177
Anthracene	0.73		0.34	J	0.36	J	46.1	1.03
Benzo(a)anthracene	3.3		1.5		1.4		51.7	4.76
Benzo(a)pyrene	2.7		1.3		1.2		48.4	3.84
Benzo(b)fluoranthene	3.9		1.8		1.7		50.4	5.59
Benzo(g,h,i)perylene	1.5		0.75		0.68		46.5	2.12
Benzo(k)fluoranthene	1.2		0.61		0.48		50.3	1.73
Chrysene	3.1		1.5		1.3		50.2	4.45
Dibenz(a,h)anthracene	0.45		0.26		0.24		36.6	0.608
Fluoranthene	5.6		2.7		2.6		46.9	7.92
Fluorene	0.22		0.1		0.13		41.6	0.307
Indeno(1,2,3-c,d)pyrene	1.8		0.83		0.81		49.4	2.57
Naphthalene	0.067		0.044		0.056		20.7	0.0846
Phenanthrene	2.8		1.4		1.5		41.1	3.87
Pyrene	5.5		2.7		2.5		47	7.79
Total LMW PAHs	7.647		3.671		3.772		45.1	10.7
Total HMW PAHs	31.197		14.921		14.182		47.8	44.3

Analyte	DU-3 (AOC #12: Former Building 410 – Power House and AOC #11: Former Building 407 – Paint Shop and Pipe Shop						% RSD	95% Upper Confidence Limit Chebychev Test
Sample ID	IINY-DU-3A-SSIS-20200318		IINY-DU-3B-SSIS-20200318		IINY-DU-3C-SSIS-20200318			
Depth (in.)	0-6		0-6		0-6			
Matrix	Surface Soil		Surface Soil		Surface Soil			
Sample Date	3/18/2020 15:45		3/18/2020 15:45		3/18/2020 15:45			
Soil pH								
pH	7.0		7.0		7.1		0.821	7.18
Mercury								
Mercury	0.14		0.13		0.11		12	0.165
Target Analyte List Metals								
Aluminum	13,000		13,000		13,000		0	13,000
Antimony	0.7		0.63		0.65		5.46	0.751
Arsenic	15		13		12		11.5	17.2
Barium	85		75		79		6.32	92.3
Beryllium	0.57		0.56		0.54		2.74	0.595
Cadmium	0.26		0.24		0.26		4.56	0.282
Calcium	3,100		8,200		7,400		44	13,100
Chromium, Total	16		15		15		3.77	16.8
Cobalt	8		7.3		7.6		4.6	8.52
Copper	28		25		27		5.73	30.5
Iron	19,000		18,000		19,000		3.09	20,100
Lead	150		120		120		13.3	174
Magnesium	4,400		7,200		6,600		24.3	9,780
Manganese	670		580		630		7.2	740
Nickel	18		16		16		6.93	19.6
Potassium	1200		1300		1400		7.69	1550
Selenium	0.45	J	0.41	J	0.39	J	7.33	0.494
Silver	0.098		0.094	U (LOQ)	0.099	U (LOQ)	2.73	0.104
Sodium	68	J	88	J	110	J	23.7	142
Thallium	0.13		0.14		0.13		4.33	0.148
Vanadium	25		23		24		4.17	26.5
Zinc	98	J	130		99	J	16.7	155
Polycyclic Aromatic Hydrocarbons								
1-Methylnaphthalene	0.015	J	0.01	J	0.0071	J	37.3	0.0208
2-Methylnaphthalene	0.017	J	0.011	J	0.0088	J	34.600	0.0229
Acenaphthene	0.036	J	0.028	J	0.015	J	40.200	0.053
Acenaphthylene	0.06	J	0.031	J	0.028	J	44.600	0.0841
Anthracene	0.14	J	0.082	J	0.052	J	49.000	0.204
Benzo(a)anthracene	0.59	J	0.35	J	0.23	J	47.000	0.851
Benzo(a)pyrene	0.61	J	0.35	J	0.25	J	46.100	0.871
Benzo(b)fluoranthene	0.93	J	0.48	J	0.37	J	50.000	1.34
Benzo(g,h,i)perylene	0.43	J	0.26	J	0.18	J	44.000	0.611
Benzo(k)fluoranthene	0.27	J	0.16	J	0.13	J	39.500	0.372
Chrysene	0.61	J	0.35	J	0.25	J	46.100	0.871
Dibenz(a,h)anthracene	0.11	J	0.064	J	0.055	J	38.600	0.151
Fluoranthene	1.1	J	0.65	J	0.43	J	47.000	1.59
Fluorene	0.044	J	0.025	J	0.015	J	52.600	0.0651
Indeno(1,2,3-c,d)pyrene	0.46	J	0.27	J	0.19	J	45.200	0.656
Naphthalene	0.023	J	0.013	J	0.012	J	38.000	0.0313
Phenanthrene	0.53	J	0.34	J	0.21	J	44.700	0.765
Pyrene	1.1	J	0.65	J	0.44	J	46.200	1.58
Total LMW PAHs	1.455		0.89		0.5779		45.600	2.09
Total HMW PAHs	6.565		3.824		2.6629		46.100	9.39

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.
DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 4a DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-4 (Previous Sampling Locations II-EA-SD-02-01 and IX407-B1)					DU-5 (AOC #3: Former Building 123 – Garage Building and Previous Sampling Location II-EA-SS-02-06)					DU-6 (Previous Sampling Locations II-EA-SS-02-09 and EA-SS-02-03)				
				Sample ID	IINY-DU-4A-SSIS-20200318	IINY-DU-4B-SSIS-20200318	IINY-DU-4C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-5A-SSIS-20200319	IINY-DU-5B-SSIS-20200319	IINY-DU-5C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-6A-SSIS-20200319	IINY-DU-6B-SSIS-20200319	IINY-DU-6C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/18/2020 14:30	3/18/2020 14:30	3/18/2020 14:30			3/19/2020 9:00	3/19/2020 9:00	3/19/2020 9:00			3/19/2020 15:00	3/19/2020 15:00	3/19/2020 15:00		
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**															
Soil pH																			
pH	N/A	N/A	N/A	N/A	6.9	7.0	7.1	1.43	7.25	7.1	7.1	7.1	1.53E-14	7.1	7.5	7.5	7.5	0	7.5
Mercury																			
Mercury	7439-97-6	1.1	4.6	mg/kg	0.29	0.34	0.23	19	0.425	0.23	0.11	0.12	43	0.321	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Target Analyte List Metals																			
Aluminum	7429-90-5	7,700	110,000	mg/kg	10,000	10,000	10,000	0	10,000	13,000	13,000	13,000	0	13,000	10,000	12,000	10,000	11	13,600
Antimony	7440-36-0	3.1	47	mg/kg	1.5	2.1	1.5	20.4	2.57	0.49	0.47	0.59	12.4	0.678	1.4	0.62	0.58	53.3	2.03
Arsenic	7440-38-2	0.68	3	mg/kg	23	30	41	29	54.2	11	11	12	5.09	12.8	10	11	8	15.8	13.5
Barium	7440-39-3	1,500	22,000	mg/kg	74	100	81	15.8	119	75	65	78	9.37	89.8	70	72	55	14.1	89
Beryllium	7440-41-7	16	230	mg/kg	0.43	0.49	0.53	10.4	0.61	0.53	0.58	0.63	8.62	0.706	0.58	0.55	0.46	11.8	0.687
Cadmium	7440-43-9	7.1	98	mg/kg	1.1	1	0.86	12.2	1.29	0.23	0.2	0.2	8.25	0.254	0.22	0.31	0.19	26	0.397
Calcium	7440-70-2	NS	NS	mg/kg	12,000	9,800	7,200	24.9	15,700	3,000 J	3,800	2,900	15.3	4,470	14,000	14,000	15,000	4.03	15,800
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	14	19	18	15.6	23.7	15	15	15	0	15	14	14	11	13.3	17.4
Cobalt	7440-48-4	2.3	35	mg/kg	6.8	7.8	7.3	6.85	8.56	7.9	7.8	7.7	1.28	8.05	7.9	7.5	6.3	11.5	9.33
Copper	7440-50-8	310	4,700	mg/kg	49	61	58	11.2	71.7	25	25	24	2.34	26.1	38	42	30	16.7	52
Iron	7439-89-6	5,500	82,000	mg/kg	19,000	20,000	19,000	2.99	20,800	20,000	20,000	19,000	2.94	21,100	16,000	19000	17000	8.81	21200
Lead	7439-92-1	400	800	mg/kg	250	360	280	19.2	440	82	76	100	14.5	117	93	130	72	29.9	172
Magnesium	7439-95-4	NS	NS	mg/kg	8,100	6,200	5,100	23.5	10,300	4,800	5,500	4,600	9.52	6,160	10,000	10,000	11,000	5.59	11,800
Manganese	7439-96-5	180	2,600	mg/kg	430	480	510	8.54	575	800	690	700	8.33	883	870	680	520	25.4	1,130
Nickel	7440-02-0	150	2,200	mg/kg	14	17	16	9.75	19.5	18	19	17	5.56	20.5	16	16	13	11.5	19.4
Potassium	2023695	NS	NS	mg/kg	1,200	1,300	1,300	4.56	1,410	1,300	1,200	1,200	4.68	1,380	1,200	1,200	1,200	0	1,200
Selenium	7782-49-2	39	580	mg/kg	0.38 J	0.5	0.58	20.7	0.74	0.27 J	0.28 J	0.31 J	7.26	0.339	0.4 J	0.36 J	0.29 J	15.9	0.49
Silver	7440-22-4	39	580	mg/kg	0.1	0.11	0.098	6.26	0.119	0.099 U (LOQ)	0.095 U (LOQ)	0.098 U (LOQ)	ND	ND	0.1	0.065 J	0.066 J	25.9	0.127
Sodium	7440-23-5	NS	NS	mg/kg	230 J	230 J	170 J	16.5	297	64 J	57 J	64 J	6.55	71.8	89 J	86 J	79 J	6.06	97.6
Thallium	7440-28-0	0.078	1.2	mg/kg	0.16	0.19	0.2	11.4	0.236	0.11	0.1	0.1	5.59	0.118	0.14	0.15	0.12	11.2	0.175
Vanadium	7440-62-2	39	580	mg/kg	20	23	23	7.87	26.4	20	19	20	2.94	21.1	23	21	17	15	28
Zinc	7440-66-6	2,300	35,000	mg/kg	280	190	150	32.2	374	74	74	79	3.82	82.9	68	79	56	17	96.6
Polycyclic Aromatic Hydrocarbons																			
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.061 J	0.19 UJ (LOD)	0.021 J	97.4	0.313	0.0056 J	0.011	0.0069 J	36	0.0149	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.069 J	0.19 UJ (LOD)	0.039 J	80.5	0.301	0.0082 J	0.013	0.0092 J	25.000	0.0165	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Acenaphthene	83-32-9	360	4,500	mg/kg	0.2 J	0.079 J	0.026 J	87.7	0.326	0.016	0.019	0.014	15.400	0.0227	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.23 J	0.25 J	0.15 J	25.2	0.343	0.016	0.019	0.024	20.500	0.0298	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Anthracene	120-12-7	1,800	23,000	mg/kg	0.39 J	0.37 J	0.18 J	37	0.605	0.068	0.052	0.062	13.300	0.081	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	1.2 J	1.3 J	0.89 J	18.9	1.67	0.4	0.25	0.3	24.100	0.509	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	1.1 J	1.1 J	1.1 J	0	1.1	0.35	0.23	0.26	22.300	0.437	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	2.9 J	2.7 J	2.1 J	16.2	3.61	0.48	0.34	0.41	17.100	0.586	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.91 J	0.86 J	0.87 J	3.01	0.947	0.22	0.15	0.17	20.000	0.271	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.92 J	0.83 J	0.66 J	16.4	1.14	0.16	0.11	0.13	18.900	0.197	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Chrysene	218-01-9	110	2,100	mg/kg	2.6 J	1.9 J	1 J	43.7	3.85	0.39	0.25	0.3	22.600	0.492	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.28 J	0.3 J	0.26 J	7.14	0.33	0.069	0.043	0.05	24.900	0.0879	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Fluoranthene	206-44-0	240	3,000	mg/kg	7.9 J	4.2 J	1.3 J	74.1	12.8	0.62	0.45	0.55	15.800	0.755	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Fluorene	86-73-7	240	3,000	mg/kg	0.27 J	0.11 J	0.043 J	82.7	0.435	0.018	0.021	0.016	13.700	0.0247	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	1 J	1.1 J	1 J	5.59	1.18	0.25	0.17	0.21	19.000	0.311	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Naphthalene	91-20-3	2	8.6	mg/kg	0.18 UJ (LOD)	0.19 UJ (LOD)	0.051 J	55.2	0.335	0.01	0.018	0.014	28.600	0.0241	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Phenanthrene	85-01-8	NS	NS	mg/kg	3.8 J	1.6 J	0.46 J	86.9	6.23	0.25	0.22	0.25	7.220	0.284	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Pyrene	129-00-0	180	2,300	mg/kg	5.7 J	3.3 J	1.3 J	64.2	8.98	0.68	0.48	0.57	17.400	0.829	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Total LMW PAHs	N/A	NS	NS	mg/kg	6.22	3.709	1.86	55.700	9.44	0.7918	0.623	0.6961	12.000	0.917	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Total HMW PAHs	N/A	NS	NS	mg/kg	25.03	17.999	11.04	38.800	35.6	3.7308	2.616	3.0761	17.800	4.55	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.
DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 4a DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-7 (Previous Sampling Location II-EA-SS-02-08)					DU-9 (AOC #8: Former Building 219 – Garage Building and AOC #9: Former Building 220 – Garage Building)					DU-10 (Previous Sampling Location II-EA-SS-02-14)			
				Sample ID	IINY-DU-7A-SSIS-20200319	IINY-DU-7B-SSIS-20200319	IINY-DU-7C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-9A-SSIS-20200319	IINY-DU-9B-SSIS-20200319	IINY-DU-9C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-10A-SSIS-20200319	IINY-DU-10B-SSIS-20200319	IINY-DU-10C-SSIS-20200319	95% Upper Confidence Limit Chebychev Test
				Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6	
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil	
				Sample Date	3/19/2020 13:45	3/19/2020 13:45	3/19/2020 13:45			3/19/2020 10:40	3/19/2020 10:40	3/19/2020 10:40			3/19/2020 11:20	3/19/2020 11:20	3/19/2020 11:20	
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit														
Soil pH																		
pH	N/A	N/A	N/A	N/A	7.4	7.4	7.5	0.777	7.58	7.1	6.9	7.0	1.43	7.25	7.1	7.1	7.2	7.28
Mercury																		
Mercury	7439-97-6	1.1	4.6	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.073	0.081	0.074	5.74	0.087	Not analyzed	Not analyzed	Not analyzed	N/A
Target Analyte List Metals																		
Aluminum	7429-90-5	7,700	110,000	mg/kg	11,000	10,000	11,000	5	12,100	10,000	11,000	11,000	5	12,100	11,000	11,000	9,300	12,900
Antimony	7440-36-0	3.1	47	mg/kg	0.75	1.6	2.5	54.1	3.82	0.44	0.42	0.44	2.66	0.462	0.41	0.41	0.35	0.477
Arsenic	7440-38-2	0.68	3	mg/kg	18	17	17	3.33	18.8	7	7.5	6.9	2.95	7.59	7.5	7.2	6.5	8.36
Barium	7440-39-3	1,500	22,000	mg/kg	110	93	110	9.41	129	61	65	59	4.95	69.4	57	58	56	59.5
Beryllium	7440-41-7	16	230	mg/kg	0.55	0.48	0.58	9.56	0.666	0.48	0.51	0.5	3.08	0.535	0.6	0.56	0.5	0.68
Cadmium	7440-43-9	7.1	98	mg/kg	0.59 J	0.48 J	0.62 J	13.1	0.749	0.23	0.22	0.26	8.8	0.289	0.17	0.19	0.19	0.212
Calcium	7440-70-2	NS	NS	mg/kg	17,000	15,000	16,000	6.25	18,500	7,600 J	5,800 J	8,700 J	19.9	11,100	14,000	14,000	17,000	19,400
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	17	15	16	6.25	18.5	13	13	13	0	13	14	14	13	15.1
Cobalt	7440-48-4	2.3	35	mg/kg	6.2	5.6	6.5	7.51	7.25	7.6	7.4	7.3	2.05	7.82	7.9	8	7.5	8.47
Copper	7440-50-8	310	4,700	mg/kg	43	60	44	19.5	73	30	28	27	5.39	32.2	23	23	23	23
Iron	7439-89-6	5,500	82,000	mg/kg	19,000	18,000	19,000	3.09	20,100	16,000	16,000	17,000 J	3.53	17,800	18,000	18,000	16,000	20,200
Lead	7439-92-1	400	800	mg/kg	690	2,000	570	73	3,080	63	58	54	7.73	69.7	31	35	37	42
Magnesium	7439-95-4	NS	NS	mg/kg	12,000	10,000	11,000	9.09	13,500	6,100 J	5,100 J	6,800 J	14.2	8,150	10,000 J	9,900	11,000	11,800
Manganese	7439-96-5	180	2,600	mg/kg	480	460	630	17.8	757	510	500	470	4.22	546	570	550	520	610
Nickel	7440-02-0	150	2,200	mg/kg	14	13	15	7.14	16.5	16	14	14	7.87	17.6	16	16	15	17.1
Potassium	2023695	NS	NS	mg/kg	1,100	1,000	1,000	5.59	1,180	1,200 J	1,300 J	1,300 J	4.56	1,410	1,400 J	1,400	1,200	1,620
Selenium	7782-49-2	39	580	mg/kg	0.38 J	0.35 J	0.43 J	10.5	0.488	0.28 J	0.28 J	0.31 J	5.97	0.334	0.27 J	0.26 J	0.3 J	0.329
Silver	7440-22-4	39	580	mg/kg	0.09 J	0.11	0.1	10	0.125	0.095 U (LOQ)	0.095 U (LOQ)	0.094 U (LOQ)	ND	ND	0.094 U (LOQ)	0.094 U (LOQ)	0.059 J	0.133
Sodium	7440-23-5	NS	NS	mg/kg	95 J	85 J	92 J	5.66	104	170 J	160 J	160 J	3.53	178	500 UJ (LOD)	83 J	88 J	826
Thallium	7440-28-0	0.078	1.2	mg/kg	0.21	0.19	0.21	5.68	0.232	0.11	0.12	0.14	12.4	0.162	0.15	0.15	0.15	0.15
Vanadium	7440-62-2	39	580	mg/kg	21	18	22	10.2	25.6	25 J	25 J	25 J	0	25	23	24	21	26.5
Zinc	7440-66-6	2,300	35,000	mg/kg	200	210	210	2.79	221	69	67	70	2.22	72.5	59	60	55	64.7
Polycyclic Aromatic Hydrocarbons																		
1-Methylnaphthalene	90-12-0	18	73	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0075 J	0.0057 J	0.0057 J	16.5	0.00892	Not analyzed	Not analyzed	Not analyzed	N/A
2-Methylnaphthalene	91-57-6	24	300	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.01	0.0082 J	0.0078 J	13.5	0.0116	Not analyzed	Not analyzed	Not analyzed	N/A
Acenaphthene	83-32-9	360	4,500	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.013	0.0078 J	0.011 J	24.7	0.0172	Not analyzed	Not analyzed	Not analyzed	N/A
Acenaphthylene	208-96-8	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.036	0.03	0.026 J	16.4	0.0433	Not analyzed	Not analyzed	Not analyzed	N/A
Anthracene	120-12-7	1,800	23,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.045	0.03	0.035 J	20.8	0.0559	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.19 J	0.13 J	0.15 J	19.5	0.234	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.21	0.15	0.17 J	17.3	0.254	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.35	0.23	0.26 J	22.3	0.437	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.15 J	0.1 J	0.11 J	22	0.187	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.12	0.078	0.086 J	23.6	0.151	Not analyzed	Not analyzed	Not analyzed	N/A
Chrysene	218-01-9	110	2,100	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.21 J	0.15 J	0.17 J	17.3	0.254	Not analyzed	Not analyzed	Not analyzed	N/A
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.038	0.027	0.028 J	19.6	0.0463	Not analyzed	Not analyzed	Not analyzed	N/A
Fluoranthene	206-44-0	240	3,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.44	0.27	0.34 J	24.4	0.565	Not analyzed	Not analyzed	Not analyzed	N/A
Fluorene	86-73-7	240	3,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.017	0.0099	0.013 J	26.8	0.0223	Not analyzed	Not analyzed	Not analyzed	N/A
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.18 J	0.12 J	0.14 J	20.8	0.224	Not analyzed	Not analyzed	Not analyzed	N/A
Naphthalene	91-20-3	2	8.6	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.013	0.011	0.0097 J	14.8	0.0154	Not analyzed	Not analyzed	Not analyzed	N/A
Phenanthrene	85-01-8	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.16 J	0.095 J	0.12 J	26.2	0.208	Not analyzed	Not analyzed	Not analyzed	N/A
Pyrene	129-00-0	180	2,300	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.41	0.27	0.3 J	22.6	0.512	Not analyzed	Not analyzed	Not analyzed	N/A
Total LMW PAHs	N/A	NS	NS	mg/kg	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A	0.4915	0.3276	0.3782	21	0.61	Not analyzed	Not analyzed	Not analyzed	N/A
Total HMW PAHs	N/A	NS	NS	mg/kg	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A	2.3795	1.5826	1.8322	21.1	2.96	Not analyzed	Not analyzed	Not analyzed	N/A

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.
DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 4a DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-11 (AOC #18: Area F – Former Coal Storage Area (Located Near Former Building 215))					DU-12 (AOC #6: Former Building 213 – Paint Locker for Building 202 and AOC #7: Former Building 215 – Garage Building)					DU-13 (AOC #1: Former Building 103 – Paint and Oil Storage and Previous Sampling Location IX 103-B1)				
				Sample ID	IINY-DU-11A-SSIS-20200330	IINY-DU-11B-SSIS-20200330	IINY-DU-11C-SSIS-20200330	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-12A-SSIS-20200330	IINY-DU-12B-SSIS-20200330	IINY-DU-12C-SSIS-20200330	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-13A-SSIS-20200330	IINY-DU-13B-SSIS-20200330	IINY-DU-13C-SSIS-20200330	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/30/2020 11:15	3/30/2020 11:15	3/30/2020 11:15			3/30/2020 12:15	3/30/2020 12:15	3/30/2020 12:15			3/30/2020 13:40	3/30/2020 13:40	3/30/2020 13:40		
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit															
Soil pH																			
pH	N/A	N/A	N/A	N/A	7.0	7.0	7.2	1.63	7.36	5.5	6.1	6.3	6.98	7.01	6.8	7.0	6.9	1.45	7.15
Mercury																			
Mercury	7439-97-6	1.1	4.6	mg/kg	0.25 J	0.15 J	0.15 J	31.5	0.329	0.26	0.26	0.21	11.9	0.316	0.28	0.31	0.36	12.8	0.418
Target Analyte List Metals																			
Aluminum	7429-90-5	7,700	110,000	mg/kg	9,700	11,000	10,000	7	11,900	11,000	11,000	11,000	0	11,000	11,000	11,000	12,000	5	12,800
Antimony	7440-36-0	3.1	47	mg/kg	0.74	0.83	0.9	9.74	1.03	0.7	0.63	0.58	9.47	0.788	1.2	1.4	2.5	41.2	3.46
Arsenic	7440-38-2	0.68	3	mg/kg	37	43	39	7.7	47.4	13	14	14	4.22	15.1	9.7	11	11	7.1	12.5
Barium	7440-39-3	1,500	22,000	mg/kg	76	79	78	1.97	81.5	84	79	79	3.58	87.9	88	86	96	5.88	103
Beryllium	7440-41-7	16	230	mg/kg	0.55	0.59	0.51	7.27	0.651	0.69	0.62	0.63	5.85	0.742	0.61	0.55	0.64	7.64	0.715
Cadmium	7440-43-9	7.1	98	mg/kg	0.37	0.36	0.37	1.57	0.381	0.38	0.33	0.35	7.12	0.417	0.74	0.77	0.75	2.03	0.792
Calcium	7440-70-2	NS	NS	mg/kg	28,000	25,000	27,000	5.73	30,500	10,000	11,000	13,000	13.5	15,200	9,200	8,500	8,900	3.96	9,750
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	15 J	15 J	15 J	0	15	15	15	15	0	15	15	14	15	3.94	16.1
Cobalt	7440-48-4	2.3	35	mg/kg	6.7	7	6.7	2.55	7.24	7.6	7.9	7.5	2.72	8.19	8.6	8.2	8.5	2.47	8.96
Copper	7440-50-8	310	4,700	mg/kg	30	37	33	10.5	42.2	51	48	47	4.28	53.9	220	600	260	58	885
Iron	7439-89-6	5,500	82,000	mg/kg	17,000 J	19,000	19,000	6.3	21,200	17,000	17,000	17,000	0	17,000	18,000	18,000	19,000	3.15	19,800
Lead	7439-92-1	400	800	mg/kg	130	110	130	9.36	152	82	83	89	4.47	94.2	190	150	160	12.5	219
Magnesium	7439-95-4	NS	NS	mg/kg	19,000	17,000	18,000	5.56	20,500	7,100	7,200	8,600	11	9,740	5,700	5,500	5,400	2.76	5,920
Manganese	7439-96-5	180	2,600	mg/kg	600	600	600	0	600	940	820	790	9.34	1,050	1,000	830	1,100	14	1,320
Nickel	7440-02-0	150	2,200	mg/kg	16	16	16	0	16	15	15	15	0	15	16	16	17	3.53	17.8
Potassium	2023695	NS	NS	mg/kg	910	960	1,000	4.71	1,070	1,100	1,100	1,100	0	1,100	1,400	1,400	1,400	0	1,400
Selenium	7782-49-2	39	580	mg/kg	0.47 J	0.61	0.57	13.1	0.731	0.51	0.54	0.24 J	38.4	0.846	0.55	0.6	0.68	10.7	0.775
Silver	7440-22-4	39	580	mg/kg	0.073 J	0.082 J	0.084 J	7.35	0.0944	0.069 J	0.069 J	0.075 J	4.88	0.0797	0.086 J	0.094 J	0.089 J	4.51	0.0998
Sodium	7440-23-5	NS	NS	mg/kg	61 J	56 J	64 J	6.7	70.5	73 J	67 J	68 J	4.64	77.4	120 J	120 J	130 J	4.68	138
Thallium	7440-28-0	0.078	1.2	mg/kg	0.19	0.22	0.22	8.25	0.254	0.15	0.14	0.15	3.94	0.161	0.16	0.16	0.18	6.93	0.196
Vanadium	7440-62-2	39	580	mg/kg	22 J	24 J	23 J	4.35	25.5	28	26	27	3.7	29.5	30	30	31	1.9	31.8
Zinc	7440-66-6	2,300	35,000	mg/kg	87	100	110	11.6	128	95	100	93	3.76	105	140	150	170	9.96	192
Polycyclic Aromatic Hydrocarbons																			
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.028 J	0.027 J	0.023 J	10.2	0.0327	0.073 J	0.044 J	0.053 J	26.2	0.094	0.38 J	0.45 J	0.26 J	26.4	0.605
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.043 J	0.044 J	0.036 J	10.6	0.052	0.098 J	0.055 J	0.053 J	37	0.133	0.43 J	0.5 J	0.31 J	23.2	0.655
Acenaphthene	83-32-9	360	4,500	mg/kg	0.02 J	0.029 J	0.015 J	33.3	0.0392	0.54 J	0.36 J	0.39 J	22.4	0.673	4.5 J	4.7 J	2.6 J	29.5	6.85
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.17 J	0.23 J	0.16 J	20.3	0.282	0.088 J	0.029 J	0.062 J	49.6	0.134	0.21 UJ (LOD)	1.3 J	0.2 UJ (LOD)	111	2.16
Anthracene	120-12-7	1,800	23,000	mg/kg	0.13 J	0.15 J	0.097 J	21.3	0.193	0.86 J	0.59 J	0.75 J	18.5	1.08	6.6 J	8 J	3.9 J	33.8	11.4
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.67 J	0.97 J	0.52 J	31.8	1.3	2.2 J	1.5 J	2.1 J	19.6	2.89	16 J	17 J	9.4 J	29.2	24.5
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.87 J	1.2 J	0.75 J	24.8	1.53	1.8 J	1.2 J	1.8 J	21.7	2.47	12 J	13 J	7.1 J	29.5	18.6
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	1.2 J	1.6 J	1.1 J	20.4	1.97	2.8 J	1.6 J	2.6 J	27.6	3.95	17 J	17 J	9.7 J	28.9	25.2
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.82 J	1.2 J	0.78 J	24.8	1.52	1.4 J	0.69 J	1.3 J	34	2.1	7.2 J	7.9 J	4.2 J	30.6	11.4
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.43 J	0.57 J	0.36 J	23.6	0.722	1.1 J	0.62 J	0.97 J	27.7	1.52	6.4 J	7.4 J	3.8 J	31.7	10.5
Chrysene	218-01-9	110	2,100	mg/kg	0.96 J	1.3 J	0.75 J	27.7	1.7	2.4 J	1.5 J	2.2 J	23.2	3.22	16 J	16 J	9.3 J	28.1	23.5
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.18 J	0.23 J	0.16 J	19	0.281	0.38 J	0.2 J	0.37 J	31.9	0.571	2.3 J	2.4 J	1.2 J	33.9	3.64
Fluoranthene	206-44-0	240	3,000	mg/kg	1.1 J	1.3 J	0.71 J	28.9	1.79	4.2 J	3.1 J	4.3 J	17.2	5.54	34 J	38 J	21 J	28.7	53.4
Fluorene	86-73-7	240	3,000	mg/kg	0.043 J	0.061 J	0.028 J	37.6	0.0856	0.44 J	0.28 J	0.32 J	24	0.556	3.2 J	3.9 J	2 J	31.7	5.45
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.78 J	1.1 J	0.74 J	22.6	1.37	1.5 J	0.76 J	1.4 J	32.9	2.23	7.9 J	9.2 J	4.9 J	30.1	12.9
Naphthalene	91-20-3	2	8.6	mg/kg	0.098 J	0.092 J	0.068 J	18.5	0.126	0.25 J	0.12 J	0.086 J	56.9	0.37	0.95 J	1.1 J	0.75 J	18.8	1.38
Phenanthrene	85-01-8	NS	NS	mg/kg	0.42 J	0.42 J	0.22 J	32.7	0.644	3.2 J	2.3 J	2.7 J	16.5	3.87	26 J	27 J	15 J	29.4	39.4
Pyrene	129-00-0	180	2,300	mg/kg	1.2 J	1.6 J	0.87 J	29.9	2.14	3.5 J	2.5 J	3.6 J	19	4.73	27 J	30 J	17 J	27.6	41.8
Total LMW PAHs	N/A	NS	NS	mg/kg	1.622	2.023	1.167	26.7	2.68	7.749	5.278	6.514	19	9.62	58.06	63.95	34.22	30.2	91.7
Total HMW PAHs	N/A	NS	NS	mg/kg	8.632	11.493	7.037	24.9	14.7	25.529	16.448	23.554	21.9	33.9	176.86	191.85	104.82	29.5	275

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.
DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 4a DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-14 (Previous Sampling Location II-EA-SS-02-13)					DU-15 (Previous Sampling Location II-EA-SS-02-15)					DU-16 (AOC #4: Former Building 124 to 128 – Sewage Disposal Plant [and Suspected Outfall])				
				Sample ID	IINY-DU-14A-SSIS-20200320	IINY-DU-14B-SSIS-20200320	IINY-DU-14C-SSIS-20200320	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-15A-SSIS-20200401	IINY-DU-15B-SSIS-20200401	IINY-DU-15C-SSIS-20200401	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-16A-SSIS-20200331	IINY-DU-16B-SSIS-20200331	IINY-DU-16C-SSIS-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/20/2020 8:30	3/20/2020 8:30	3/20/2020 8:30			4/1/2020 8:20	4/1/2020 8:20	4/1/2020 8:20			3/31/2020 9:50	3/31/2020 9:50	3/31/2020 9:50		
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**															
Soil pH																			
pH	N/A	N/A	N/A	N/A	6.7	6.9	7.0	2.22	7.25	6.2	6.3	6.6	3.27	6.89	6.9	6.9	7.4	4.09	7.79
Mercury																			
Mercury	7439-97-6	1.1	4.6	mg/kg	0.16	0.18	0.19	8.65	0.215	0.14	0.21	0.29	35.2	0.402	0.097	0.096	0.096	0.599	0.0978
Target Analyte List Metals																			
Aluminum	7429-90-5	7,700	110,000	mg/kg	10,000	11,000	10,000	6	11,800	11,000	11,000	11,000	0	11,000	15,000	14,000	13,000	7	16,500
Antimony	7440-36-0	3.1	47	mg/kg	0.6	0.56	0.59	3.57	0.636	1.1	0.7	0.7	27.7	1.41	0.44	0.4	0.42	4.76	0.47
Arsenic	7440-38-2	0.68	3	mg/kg	16	18	17	5.88	19.5	7	6.5	7.1	4.68	7.68	7.1	6.8	7.2	2.96	7.56
Barium	7440-39-3	1,500	22,000	mg/kg	76	81	78	3.21	84.7	58	61	62	3.45	65.6	61	56	59	4.29	65
Beryllium	7440-41-7	16	230	mg/kg	0.53	0.57	0.55	3.64	0.6	0.47	0.5	0.47	3.61	0.524	0.6	0.6	0.53	7.01	0.678
Cadmium	7440-43-9	7.1	98	mg/kg	0.28	0.27	0.28	2.09	0.291	0.58	0.38	0.58	22.5	0.804	0.25	0.33	0.27	14.7	0.388
Calcium	7440-70-2	NS	NS	mg/kg	9,500 J	8,900 J	6,800 J	16.9	12,000	7,100	7,500	9,900	18.5	12,000	8,000	7,600	7,200	5.26	8,610
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	13	14	14	4.22	15.1	15	15	15	0	15	15	15	14	3.94	16.1
Cobalt	7440-48-4	2.3	35	mg/kg	6.8	6.9	7.3	3.78	7.67	6.8	6.7	6.8	0.853	6.91	8.5	8	8.4	3.19	8.97
Copper	7440-50-8	310	4,700	mg/kg	23	26	25	6.19	28.5	30 J	31 J	33 J	4.88	35.2	38	33	39	8.77	44.8
Iron	7439-89-6	5,500	82,000	mg/kg	16,000	17,000	16,000 J	3.53	17,800	19,000	18,000	18,000	3.15	19,800	22,000	20,000	21,000	4.76	23,500
Lead	7439-92-1	400	800	mg/kg	96	120	110	11.1	139	130	130	120	4.56	141	38	42	46	9.52	52.1
Magnesium	7439-95-4	NS	NS	mg/kg	7,100 J	6,800 J	5,500 J	13.2	8,610	6,000	5,900	7,700	15.5	9,080	6,500	6,200	6,100	3.32	6,790
Manganese	7439-96-5	180	2,600	mg/kg	670	720	700	3.61	760	410	480	430	8.19	531	450	430	440	2.27	465
Nickel	7440-02-0	150	2,200	mg/kg	15	16	16	3.69	17.1	14	14	15	4.03	15.8	16	16	16	0	16
Potassium	2023695	NS	NS	mg/kg	980	1,000	1,000	1.16	1,020	1,200	1,200	1,200	0	1,200	1,700	1,600	1,400	9.75	1,950
Selenium	7782-49-2	39	580	mg/kg	0.38 J	0.38 J	0.4 J	2.99	0.416	0.52	0.62	0.47 J	14.2	0.729	0.4 J	0.4 J	0.35 J	7.53	0.456
Silver	7440-22-4	39	580	mg/kg	0.08 J	0.063 J	0.087 J	16.1	0.108	0.079 J	0.089	0.073 J	10.1	0.101	0.055 J	0.063 J	0.057 J	7.14	0.0688
Sodium	7440-23-5	NS	NS	mg/kg	72 J	70 J	73 J	2.13	75.5	75 J	81 J	77 J	3.93	85.4	440 J	360 J	330 J	15.1	520
Thallium	7440-28-0	0.078	1.2	mg/kg	0.11	0.13	0.12	8.33	0.145	0.16	0.15	0.16	3.69	0.171	0.13	0.12	0.12	4.68	0.138
Vanadium	7440-62-2	39	580	mg/kg	20	20	20	0	20	25	26	25	2.28	26.8	28	30	30	3.94	32.2
Zinc	7440-66-6	2,300	35,000	mg/kg	74	74	78	3.07	81.1	250	120	120	46	352	70	71	66	3.83	75.7
Polycyclic Aromatic Hydrocarbons																			
1-Methylnaphthalene	90-12-0	18	73	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.026 J	0.028	0.0077 J	54.4	0.0487
2-Methylnaphthalene	91-57-6	24	300	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.033 J	0.041	0.0082 J	62.4	0.0704
Acenaphthene	83-32-9	360	4,500	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.15 J	0.14	0.036	58.1	0.268
Acenaphthylene	208-96-8	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.032 J	0.034	0.028	9.75	0.039
Anthracene	120-12-7	1,800	23,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.18 J	0.16	0.063	46.6	0.292
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.5 J	0.44	0.21	39.9	0.769
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.47 J	0.43	0.24	32.3	0.689
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.71 J	0.65	0.33	36.3	1.08
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.31 J	0.29	0.15	34.9	0.469
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.26 J	0.23	0.12	36.3	0.389
Chrysene	218-01-9	110	2,100	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.49 J	0.44	0.21	39.3	0.756
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.1 J	0.085	0.042	39.8	0.151
Fluoranthene	206-44-0	240	3,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	1 J	0.96	0.41	41.7	1.62
Fluorene	86-73-7	240	3,000	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.1 J	0.1	0.027	55.7	0.182
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.38 J	0.35	0.19	33.3	0.564
Naphthalene	91-20-3	2	8.6	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.064 J	0.089	0.012	71.4	0.154
Phenanthrene	85-01-8	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.74 J	0.66	0.2	54.6	1.27
Pyrene	129-00-0	180	2,300	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.86 J	0.8	0.35	41.6	1.37
Total LMW PAHs	N/A	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	1.825	1.692	0.5919	49.4	3.07
Total HMW PAHs	N/A	NS	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	6.045	5.567	2.4939	41	9.55

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.
DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 4b DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-1 (AOC #17: Area E – Former Coal Trestle and Storage Area [Located Near Former Building 410] and AOC #10: Former Building 406 – Tin, Electrical, and Annealing Building Shop)					DU-2 (AOC #17: Area E – Former Coal Trestle and Storage Area [Located Near Former Building 410])					DU-3 (AOC #12: Former Building 410 – Power House and AOC #11: Former Building 407 – Paint Shop and Pipe Shop				
			Sample ID	IINY-DU-1A-SSIS-20200318	IINY-DU-1B-SSIS-20200318	IINY-DU-1C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-2A-SSIS-20200319	IINY-DU-2B-SSIS-20200319	IINY-DU-2C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-3A-SSIS-20200318	IINY-DU-3B-SSIS-20200318	IINY-DU-3C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
			Sample Date	3/18/2020 15:00	3/18/2020 15:00	3/18/2020 15:00			3/19/2020 8:15	3/19/2020 8:15	3/19/2020 8:15			3/18/2020 15:45	3/18/2020 15:45	3/18/2020 15:45		
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit															
Soil pH																		
pH	N/A	N/A	N/A	5.6	6.5	6.8	9.91	7.87	5.6	6.6	6.8	10.2	7.95	7.0	7.0	7.1	0.821	7.18
Mercury																		
Mercury	7439-97-6	0.013	mg/kg	**0.084**	**0.08**	**0.078**	4	**0.0884**	**0.091**	**0.092**	**0.11**	11	**0.125**	**0.14**	**0.13**	**0.11**	12	**0.165**
Target Analyte List Metals																		
Aluminum	7429-90-5	NS	mg/kg	11,000	11,000	11,000	0	11,000	13,000	13,000	13,000	0	13,000	13,000	13,000	13,000	0	13,000
Antimony	7440-36-0	0.27	mg/kg	**0.52**	**0.51**	**0.47**	5.29	**0.567**	**0.61**	**0.62**	**0.68**	5.95	**0.732**	**0.7**	**0.63**	**0.65**	5.46	**0.751**
Arsenic	7440-38-2	6.8	mg/kg	**11**	**12**	**8.9** J	14.9	**14.6**	**14**	**13**	**14**	4.22	**15.1**	**15**	**13**	**12**	11.5	**17.2**
Barium	7440-39-3	110	mg/kg	71	66	65	4.77	75.4	68	74	79	7.48	87.5	85	75	79	6.32	92.3
Beryllium	7440-41-7	2.5	mg/kg	0.52	0.5	0.55	4.81	0.587	0.54	0.55	0.65	10.5	0.733	0.57	0.56	0.54	2.74	0.595
Cadmium	7440-43-9	0.36	mg/kg	0.35	0.26	0.24	20.7	**0.431**	0.2	0.23	0.25	11.1	0.29	0.26	0.24	0.26	4.56	0.282
Calcium	7440-70-2	NS	mg/kg	3,700	5,100	4,800	16.3	6,390	13,000	11,000	7,800	24.7	17,200	3,100	8,200	7,400	44	13,100
Chromium, Total	7440-47-3	23	mg/kg	14	13	13	4.33	14.8	15	15	15	0	15	16	15	15	3.77	16.8
Cobalt	7440-48-4	13	mg/kg	7	6.8	7.5	5.08	8.01	7.6	7.4	7.7	2.02	7.95	8	7.3	7.6	4.6	8.52
Copper	7440-50-8	28	mg/kg	21	22	21	2.71	22.8	27	26	28	3.7	**29.5**	28	25	27	5.73	**30.5**
Iron	7439-89-6	NS	mg/kg	16,000	16,000	15,000	3.69	17,100	19,000	18,000	19,000	3.09	20,100	19,000	18,000	19,000	3.09	20,100
Lead	7439-92-1	11	mg/kg	**84**	**57**	**52**	26.8	**108**	**78**	**93**	**110**	17.1	**134**	**150**	**120**	**120**	13.3	**174**
Magnesium	7439-95-4	NS	mg/kg	3,800	4,700	4,300	10.6	5,400	10,000	9,000	7,300	15.6	12,200	4,400	7,200	6,600	24.3	9,780
Manganese	7439-96-5	220	mg/kg	**470**	**470**	**480**	1.22	**488**	**620**	**660**	**640**	3.12	**690**	**670**	**580**	**630**	7.2	**740**
Nickel	7440-02-0	38	mg/kg	14	14	14	0	14	17	16	18	5.88	19.5	18	16	16	6.93	19.6
Potassium	2023695	NS	mg/kg	1,200	1,200	1,200	0	1,200	1,400	1,300	1,200	7.69	1,550	1,200	1,300	1,400	7.69	1,550
Selenium	7782-49-2	0.52	mg/kg	0.44 J	0.32 J	0.31 J	20.3	**0.539**	0.31 J	0.33 J	0.32 J	3.13	0.345	0.45 J	0.41 J	0.39 J	7.33	0.494
Silver	7440-22-4	4.2	mg/kg	0.093 U (LOQ)	0.099 U (LOQ)	0.099 U (LOQ)	ND	ND	0.097 U (LOQ)	0.097 U (LOQ)	0.1 U (LOQ)	ND	ND	0.098	0.094 U (LOQ)	0.099 U (LOQ)	2.73	0.104
Sodium	7440-23-5	NS	mg/kg	95 J	100 J	110 J	7.51	121	75 J	75 J	71 J	3.13	79.5	68 J	88 J	110 J	23.7	142
Thallium	7440-28-0	0.05	mg/kg	**0.14**	**0.12**	**0.12**	9.12	**0.156**	**0.13**	**0.13**	**0.13**	0	**0.13**	**0.13**	**0.14**	**0.13**	4.33	**0.148**
Vanadium	7440-62-2	7.8	mg/kg	**23**	**21**	**22**	4.55	**24.5**	**21**	**21**	**23**	5.33	**24.6**	**25**	**23**	**24**	4.17	**26.5**
Zinc	7440-66-6	46	mg/kg	**92** J	**76** J	**67** J	16.2	**110**	**72**	**74**	**80**	5.53	**85.8**	**98** J	**130**	**99** J	16.7	**155**
Polycyclic Aromatic Hydrocarbons																		
1-Methylnaphthalene	90-12-0	29	mg/kg	0.0058 J	0.0044 J	0.0036 J	24.2	0.0074	0.075	0.036	0.046	38.7	0.103	0.015 J	0.01 J	0.0071 J	37.3	0.0208
2-Methylnaphthalene	91-57-6	29	mg/kg	0.0098 J	0.0062 J	0.0053 J	33.500	0.0131	0.055	0.031	0.04	28.9	0.0725	0.017 J	0.011 J	0.0088 J	34.600	0.0229
Acenaphthene	83-32-9	29	mg/kg	0.007 J	0.0068 J	0.0054 J	13.600	0.00859	0.26	0.12	0.15	41.7	0.362	0.036 J	0.028 J	0.015 J	40.200	0.053
Acenaphthylene	208-96-8	29	mg/kg	0.041 J	0.027 J	0.026 J	26.800	0.0524	0.14	0.1	0.09	24.1	0.177	0.06 J	0.031 J	0.028 J	44.600	0.0841
Anthracene	120-12-7	29	mg/kg	0.032 J	0.028 J	0.025 J	12.400	0.0372	0.73	0.34 J	0.36 J	46.1	1.03	0.14 J	0.082 J	0.052 J	49.000	0.204
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.15 J	0.13 J	0.11 J	15.400	0.18	**3.3**	**1.5**	**1.4**	51.7	**4.76**	0.59 J	0.35 J	0.23 J	47.000	0.851
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.17 J	0.15 J	0.13 J	13.300	0.2	**2.7**	**1.3**	**1.2**	48.4	**3.84**	0.61 J	0.35 J	0.25 J	46.100	0.871
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	0.27 J	0.23 J	0.2 J	15.100	0.322	**3.9**	**1.8**	**1.7**	50.4	**5.59**	0.93 J	0.48 J	0.37 J	50.000	**1.34**
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.13 J	0.12 J	0.1 J	13.100	0.155	**1.5**	0.75	0.68	46.5	**2.12**	0.43 J	0.26 J	0.18 J	44.000	0.611
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.092 J	0.076 J	0.065 J	17.500	0.112	**1.2**	0.61	0.48	50.3	**1.73**	0.27 J	0.16 J	0.13 J	39.500	0.372
Chrysene	218-01-9	1.1	mg/kg	0.17 J	0.14 J	0.12 J	17.600	0.207	**3.1**	**1.5**	**1.3**	50.2	**4.45**	0.61 J	0.35 J	0.25 J	46.100	0.871
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.035 J	0.03 J	0.026 J	14.900	0.0417	0.45	0.26	0.24	36.6	0.608	0.11 J	0.064 J	0.055 J	38.600	0.151
Fluoranthene	206-44-0	1.1	mg/kg	0.26 J	0.24 J	0.2 J	13.100	0.31	**5.6**	**2.7**	**2.6**	46.9	**7.92**	1.1 J	0.65 J	0.43 J	47.000	**1.59**
Fluorene	86-73-7	29	mg/kg	0.009 J	0.0077 J	0.008 J	8.270	0.00995	0.22	0.1	0.13	41.6	0.307	0.044 J	0.025 J	0.015 J	52.600	0.0651
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.15 J	0.14 J	0.12 J	11.200	0.175	**1.8**	0.83	0.81	49.4	**2.57**	0.46 J	0.27 J	0.19 J	45.200	0.656
Naphthalene	91-20-3	29	mg/kg	0.011 J	0.0075 J	0.0063 J	29.500	0.0144	0.067	0.044	0.056	20.7	0.0846	0.023 J	0.013 J	0.012 J	38.000	0.0313
Phenanthrene	85-01-8	29	mg/kg	0.084 J	0.09 J	0.074 J	9.780	0.103	2.8	1.4	1.5	41.1	3.87	0.53 J	0.34 J	0.21 J	44.700	0.765
Pyrene	129-00-0	1.1	mg/kg	0.26 J	0.24 J	0.2 J	13.100	0.31	**5.5**	**2.7**	**2.5**	47	**7.79**	1.1 J	0.65 J	0.44 J	46.200	**1.58**
Total LMW PAHs	N/A	29	mg/kg	0.3496	0.3076	0.2636	14	0.415	7.647	3.671	3.772	**45.1**	10.7	1.455	0.89	0.5779	45.600	2.09
Total HMW PAHs	N/A	1.1	mg/kg	**1.7766**	**1.5636**	**1.3346**	14.2	**2.11**	**31.197**	**14.921**	**14.182**	47.8	**44.3**	**6.565**	**3.824**	**2.6629**	46.100	**9.39**

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.

Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 4b DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-4 (Previous Sampling Locations II-EA-SD-02-01 and IX407-B1)					DU-5 (AOC #3: Former Building 123 – Garage Building and Previous Sampling Location II-EA-SS-02-06)					DU-6 (Previous Sampling Locations II-EA-SS-02-09 and EA-SS-02-03)				
			Sample ID	IINY-DU-4A-SSIS-20200318	IINY-DU-4B-SSIS-20200318	IINY-DU-4C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-5A-SSIS-20200319	IINY-DU-5B-SSIS-20200319	IINY-DU-5C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-6A-SSIS-20200319	IINY-DU-6B-SSIS-20200319	IINY-DU-6C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
			Sample Date	3/18/2020 14:30	3/18/2020 14:30	3/18/2020 14:30			3/19/2020 9:00	3/19/2020 9:00	3/19/2020 9:00			3/19/2020 15:00	3/19/2020 15:00	3/19/2020 15:00		
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit															
Soil pH																		
pH	N/A	N/A	N/A	6.9	7.0	7.1	1.43	7.25	7.1	7.1	7.1	1.53E-14	7.1	7.5	7.5	7.5	0	7.5
Mercury																		
Mercury	7439-97-6	0.013	mg/kg	**0.29**	**0.34**	**0.23**	19	**0.425**	**0.23**	**0.11**	**0.12**	43	**0.321**	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Target Analyte List Metals																		
Aluminum	7429-90-5	NS	mg/kg	10,000	10,000	10,000	0	10,000	13,000	13,000	13,000	0	13,000	10,000	12,000	10,000	11	13,600
Antimony	7440-36-0	0.27	mg/kg	**1.5**	**2.1**	**1.5**	20.4	**2.57**	**0.49**	**0.47**	**0.59**	12.4	**0.678**	**1.4**	**0.62**	**0.58**	53.3	**2.03**
Arsenic	7440-38-2	6.8	mg/kg	**23**	**30**	**41**	29	**54.2**	**11**	**11**	**12**	5.09	**12.8**	**10**	**11**	**8**	15.8	**13.5**
Barium	7440-39-3	110	mg/kg	74	100	81	15.8	**119**	75	65	78	9.37	89.8	70	72	55	14.1	89
Beryllium	7440-41-7	2.5	mg/kg	0.43	0.49	0.53	10.4	0.61	0.53	0.58	0.63	8.62	0.706	0.58	0.55	0.46	11.8	0.687
Cadmium	7440-43-9	0.36	mg/kg	**1.1**	**1**	**0.86**	12.2	**1.29**	0.23	0.2	0.2	8.25	0.254	0.22	0.31	0.19	26	0.397
Calcium	7440-70-2	NS	mg/kg	12,000	9,800	7,200	24.9	15,700	3,000 J	3,800	2,900	15.3	4,470	14,000	14,000	15,000	4.03	15,800
Chromium, Total	7440-47-3	23	mg/kg	14	19	18	15.6	**23.7**	15	15	15	0	15	14	14	11	13.3	17.4
Cobalt	7440-48-4	13	mg/kg	6.8	7.8	7.3	6.85	8.56	7.9	7.8	7.7	1.28	8.05	7.9	7.5	6.3	11.5	9.33
Copper	7440-50-8	28	mg/kg	**49**	**61**	**58**	11.2	**71.7**	25	25	24	2.34	26.1	**38**	**42**	**30**	16.7	**52**
Iron	7439-89-6	NS	mg/kg	19,000	20,000	19,000	2.99	20,800	20,000	20,000	19,000	2.94	21,100	16,000	19000	17000	8.81	21200
Lead	7439-92-1	11	mg/kg	**250**	**360**	**280**	19.2	**440**	**82**	**76**	**100**	14.5	**117**	**93**	**130**	**72**	29.9	**172**
Magnesium	7439-95-4	NS	mg/kg	8,100	6,200	5,100	23.5	10,300	4,800	5,500	4,600	9.52	6,160	10,000	10,000	11,000	5.59	11,800
Manganese	7439-96-5	220	mg/kg	**430**	**480**	**510**	8.54	**575**	**800**	**690**	**700**	8.33	**883**	**870**	**680**	**520**	25.4	**1,130**
Nickel	7440-02-0	38	mg/kg	14	17	16	9.75	19.5	18	19	17	5.56	20.5	16	16	13	11.5	19.4
Potassium	2023695	NS	mg/kg	1,200	1,300	1,300	4.56	1,410	1,300	1,200	1,200	4.68	1,380	1,200	1,200	1,200	0	1,200
Selenium	7782-49-2	0.52	mg/kg	0.38 J	0.5	**0.58**	20.7	**0.74**	0.27 J	0.28 J	0.31 J	7.26	0.339	0.4 J	0.36 J	0.29 J	15.9	0.49
Silver	7440-22-4	4.2	mg/kg	0.1	0.11	0.098	6.26	0.119	0.099 U (LOQ)	0.095 U (LOQ)	0.098 U (LOQ)	ND	ND	0.1	0.065 J	0.066 J	25.9	0.127
Sodium	7440-23-5	NS	mg/kg	230 J	230 J	170 J	16.5	297	64 J	57 J	64 J	6.55	71.8	89 J	86 J	79 J	6.06	97.6
Thallium	7440-28-0	0.05	mg/kg	**0.16**	**0.19**	**0.2**	11.4	**0.236**	**0.11**	**0.1**	**0.1**	5.59	**0.118**	**0.14**	**0.15**	**0.12**	11.2	**0.175**
Vanadium	7440-62-2	7.8	mg/kg	**20**	**23**	**23**	7.87	**26.4**	**20**	**19**	**20**	2.94	**21.1**	**23**	**21**	**17**	15	**28**
Zinc	7440-66-6	46	mg/kg	**280**	**190**	**150**	32.2	**374**	**74**	**74**	**79**	3.82	**82.9**	**68**	**79**	**56**	17	**96.6**
Polycyclic Aromatic Hydrocarbons																		
1-Methylnaphthalene	90-12-0	29	mg/kg	0.061 J	0.19 UJ (LOD)	0.021 J	97.4	0.313	0.0056 J	0.011	0.0069 J	36	0.0149	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
2-Methylnaphthalene	91-57-6	29	mg/kg	0.069 J	0.19 UJ (LOD)	0.039 J	80.5	0.301	0.0082 J	0.013	0.0092 J	25.000	0.0165	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Acenaphthene	83-32-9	29	mg/kg	0.2 J	0.079 J	0.026 J	87.7	0.326	0.016	0.019	0.014	15.400	0.0227	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Acenaphthylene	208-96-8	29	mg/kg	0.23 J	0.25 J	0.15 J	25.2	0.343	0.016	0.019	0.024	20.500	0.0298	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Anthracene	120-12-7	29	mg/kg	0.39 J	0.37 J	0.18 J	37	0.605	0.068	0.052	0.062	13.300	0.081	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(a)anthracene	56-55-3	1.1	mg/kg	**1.2** J	**1.3** J	0.89 J	18.9	**1.67**	0.4	0.25	0.3	24.100	0.509	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(a)pyrene	50-32-8	1.1	mg/kg	1.1 J	1.1 J	1.1 J	0	1.1	0.35	0.23	0.26	22.300	0.437	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	**2.9** J	**2.7** J	**2.1** J	16.2	**3.61**	0.48	0.34	0.41	17.100	0.586	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.91 J	0.86 J	0.87 J	3.01	0.947	0.22	0.15	0.17	20.000	0.271	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.92 J	0.83 J	0.66 J	16.4	**1.14**	0.16	0.11	0.13	18.900	0.197	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Chrysene	218-01-9	1.1	mg/kg	**2.6** J	**1.9** J	1 J	43.7	**3.85**	0.39	0.25	0.3	22.600	0.492	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.28 J	0.3 J	0.26 J	7.14	0.33	0.069	0.043	0.05	24.900	0.0879	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Fluoranthene	206-44-0	1.1	mg/kg	**7.9** J	**4.2** J	**3.3** J	74.1	**12.8**	0.62	0.45	0.55	15.800	0.755	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Fluorene	86-73-7	29	mg/kg	0.27 J	0.11 J	0.043 J	82.7	0.435	0.018	0.021	0.016	13.700	0.0247	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	1 J	1.1 J	1 J	5.59	**1.18**	0.25	0.17	0.21	19.000	0.311	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Naphthalene	91-20-3	29	mg/kg	0.18 UJ (LOD)	0.19 UJ (LOD)	0.051 J	55.2	0.335	0.01	0.018	0.014	28.600	0.0241	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Phenanthrene	85-01-8	29	mg/kg	3.8 J	1.6 J	0.46 J	86.9	6.23	0.25	0.22	0.25	7.220	0.284	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Pyrene	129-00-0	1.1	mg/kg	**5.7** J	**3.3** J	**1.3** J	64.2	**8.98**	0.68	0.48	0.57	17.400	0.829	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Total LMW PAHs	N/A	29	mg/kg	6.22	3.709	1.86	55.700	9.44	0.7918	0.623	0.6961	12.000	0.917	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A
Total HMW PAHs	N/A	1.1	mg/kg	**25.03**	**17.999**	**11.04**	38.800	**35.6**	**3.7308**	**2.616**	**3.0761**	17.800	**4.55**	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.

Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 4b DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-7 (Previous Sampling Location II-EA-SS-02-08)					DU-9 (AOC #8: Former Building 219 – Garage Building and AOC #9: Former Building 220 – Garage Building)					DU-10 (Previous Sampling Location II-EA-SS-02-14)			
			Sample ID	IINY-DU-7A-SSIS-20200319	IINY-DU-7B-SSIS-20200319	IINY-DU-7C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-9A-SSIS-20200319	IINY-DU-9B-SSIS-20200319	IINY-DU-9C-SSIS-20200319	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-10A-SSIS-20200319	IINY-DU-10B-SSIS-20200319	IINY-DU-10C-SSIS-20200319	95% Upper Confidence Limit Chebychev Test
			Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6	
			Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil	
			Sample Date	3/19/2020 13:45	3/19/2020 13:45	3/19/2020 13:45			3/19/2020 10:40	3/19/2020 10:40	3/19/2020 10:40			3/19/2020 11:20	3/19/2020 11:20	3/19/2020 11:20	
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**														
Soil pH																	
pH	N/A	N/A	N/A	7.4	7.4	7.5	0.777	7.58	7.1	6.9	7.0	1.43	7.25	7.1	7.1	7.2	7.28
Mercury																	
Mercury	7439-97-6	0.013	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	**0.073**	**0.081**	**0.074**	5.74	**0.087**	Not analyzed	Not analyzed	Not analyzed	N/A
Target Analyte List Metals																	
Aluminum	7429-90-5	NS	mg/kg	11,000	10,000	11,000	5	12,100	10,000	11,000	11,000	5	12,100	11,000	11,000	9,300	12,900
Antimony	7440-36-0	0.27	mg/kg	**0.75**	**1.6**	**2.5**	54.1	**3.82**	**0.44**	**0.42**	**0.44**	2.66	**0.462**	**0.41**	**0.41**	**0.35**	**0.477**
Arsenic	7440-38-2	6.8	mg/kg	**18**	**17**	**17**	3.33	**18.8**	**7**	**7.3**	**6.9**	2.95	**7.59**	**7.5**	**7.2**	6.5	**8.36**
Barium	7440-39-3	110	mg/kg	110	93	110	9.41	**129**	61	65	59	4.95	69.4	57	58	56	59.5
Beryllium	7440-41-7	2.5	mg/kg	0.55	0.48	0.58	9.56	0.666	0.48	0.51	0.5	3.08	0.535	0.6	0.56	0.5	0.68
Cadmium	7440-43-9	0.36	mg/kg	**0.59** J	**0.48** J	**0.62** J	13.1	**0.749**	0.23	0.22	0.26	8.8	0.289	0.17	0.19	0.19	**0.212**
Calcium	7440-70-2	NS	mg/kg	17,000	15,000	16,000	6.25	18,500	7,600 J	5,800 J	8,700 J	19.9	11,100	14,000	14,000	17,000	19,400
Chromium, Total	7440-47-3	23	mg/kg	17	15	16	6.25	18.5	13	13	13	0	13	14	14	13	15.1
Cobalt	7440-48-4	13	mg/kg	6.2	5.6	6.5	7.51	7.25	7.6	7.4	7.3	2.05	7.82	7.9	8	7.5	8.47
Copper	7440-50-8	28	mg/kg	**43**	**60**	**44**	19.5	**73**	**30**	28	27	5.39	**32.2**	23	23	23	23
Iron	7439-89-6	NS	mg/kg	19,000	18,000	19,000	3.09	20,100	16,000	16,000	17,000 J	3.53	17,800	18,000	18,000	16,000	20,200
Lead	7439-92-1	11	mg/kg	**690**	**2,000**	**570**	73	**3,080**	**63**	**58**	**54**	7.73	**69.7**	**31**	**35**	**37**	**42**
Magnesium	7439-95-4	NS	mg/kg	12,000	10,000	11,000	9.09	13,500	6,100 J	5,100 J	6,800 J	14.2	8,150	10,000 J	9,900	11,000	11,800
Manganese	7439-96-5	220	mg/kg	**480**	**460**	**630**	17.8	**757**	**510**	**500**	**470**	4.22	**546**	**570**	**550**	**520**	**610**
Nickel	7440-02-0	38	mg/kg	14	13	15	7.14	16.5	16	14	14	7.87	17.6	16	16	15	17.1
Potassium	2023695	NS	mg/kg	1,100	1,000	1,000	5.59	1,180	1,200 J	1,300 J	1,300 J	4.56	1,410	1,400 J	1,400	1,200	1,620
Selenium	7782-49-2	0.52	mg/kg	0.38 J	0.35 J	0.43 J	10.5	0.488	0.28 J	0.28 J	0.31 J	5.97	0.334	0.27 J	0.26 J	0.3 J	0.329
Silver	7440-22-4	4.2	mg/kg	0.09 J	0.11	0.1	10	0.125	0.095 U (LOQ)	0.095 U (LOQ)	0.094 U (LOQ)	ND	ND	0.094 U (LOQ)	0.094 U (LOQ)	0.059 J	0.133
Sodium	7440-23-5	NS	mg/kg	95 J	85 J	92 J	5.66	104	170 J	160 J	160 J	3.53	178	500 UJ (LOD)	83 J	88 J	826
Thallium	7440-28-0	0.05	mg/kg	**0.21**	**0.19**	**0.21**	5.68	**0.232**	**0.11**	**0.12**	**0.14**	12.4	**0.162**	**0.15**	**0.15**	**0.15**	**0.15**
Vanadium	7440-62-2	7.8	mg/kg	**21**	**18**	**22**	10.2	**25.6**	**25** J	**25** J	**25** J	0	**25**	**23**	**24**	**21**	**26.5**
Zinc	7440-66-6	46	mg/kg	**200**	**210**	**210**	2.79	**221**	**69**	**67**	**70**	2.22	**72.5**	**59**	**60**	**55**	**64.7**
Polycyclic Aromatic Hydrocarbons																	
1-Methylnaphthalene	90-12-0	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0075 J	0.0057 J	0.0057 J	16.5	0.00892	Not analyzed	Not analyzed	Not analyzed	N/A
2-Methylnaphthalene	91-57-6	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.01	0.0082 J	0.0078 J	13.5	0.0116	Not analyzed	Not analyzed	Not analyzed	N/A
Acenaphthene	83-32-9	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.013	0.0078 J	0.011 J	24.7	0.0172	Not analyzed	Not analyzed	Not analyzed	N/A
Acenaphthylene	208-96-8	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.036	0.03	0.026 J	16.4	0.0433	Not analyzed	Not analyzed	Not analyzed	N/A
Anthracene	120-12-7	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.045	0.03	0.035 J	20.8	0.0559	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(a)anthracene	56-55-3	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.19 J	0.13 J	0.15 J	19.5	0.234	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(a)pyrene	50-32-8	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.21	0.15	0.17 J	17.3	0.254	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.35	0.23	0.26 J	22.3	0.437	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.15 J	0.1 J	0.11 J	22	0.187	Not analyzed	Not analyzed	Not analyzed	N/A
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.12	0.078	0.086 J	23.6	0.151	Not analyzed	Not analyzed	Not analyzed	N/A
Chrysene	218-01-9	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.21 J	0.15 J	0.17 J	17.3	0.254	Not analyzed	Not analyzed	Not analyzed	N/A
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.038	0.027	0.028 J	19.6	0.0463	Not analyzed	Not analyzed	Not analyzed	N/A
Fluoranthene	206-44-0	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.44	0.27	0.34 J	24.4	0.565	Not analyzed	Not analyzed	Not analyzed	N/A
Fluorene	86-73-7	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.017	0.0099	0.013 J	26.8	0.0223	Not analyzed	Not analyzed	Not analyzed	N/A
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.18 J	0.12 J	0.14 J	20.8	0.224	Not analyzed	Not analyzed	Not analyzed	N/A
Naphthalene	91-20-3	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.013	0.011	0.0097 J	14.8	0.0154	Not analyzed	Not analyzed	Not analyzed	N/A
Phenanthrene	85-01-8	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.16 J	0.095 J	0.12 J	26.2	0.208	Not analyzed	Not analyzed	Not analyzed	N/A
Pyrene	129-00-0	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.41	0.27	0.3 J	22.6	0.512	Not analyzed	Not analyzed	Not analyzed	N/A
Total LMW PAHs	N/A	29	mg/kg	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A	0.4915	0.3276	0.3782	21	0.61	Not analyzed	Not analyzed	Not analyzed	N/A
Total HMW PAHs	N/A	1.1	mg/kg	Not Analyzed	Not Analyzed	Not Analyzed	N/A	N/A	**2.3795**	**1.5826**	**1.8322**	21.1	**2.96**	Not analyzed	Not analyzed	Not analyzed	N/A

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
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PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 4b DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-11 (AOC #18: Area F – Former Coal Storage Area (Located Near Former Building 215])								DU-12 (AOC #6: Former Building 213 – Paint Locker for Building 202 and AOC #7: Former Building 215 – Garage Building)						DU-13 (AOC #1: Former Building 103 – Paint and Oil Storage and Previous Sampling Location IX 103-B1)					
			Sample ID	IINY-DU-11A-SSIS-20200330		IINY-DU-11B-SSIS-20200330		IINY-DU-11C-SSIS-20200330		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-12A-SSIS-20200330		IINY-DU-12B-SSIS-20200330		IINY-DU-12C-SSIS-20200330		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-13A-SSIS-20200330		IINY-DU-13B-SSIS-20200330		IINY-DU-13C-SSIS-20200330		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in.)	0-6		0-6		0-6				0-6		0-6		0-6				0-6		0-6		0-6			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
			Sample Date	3/30/2020 11:15		3/30/2020 11:15		3/30/2020 11:15				3/30/2020 12:15		3/30/2020 12:15		3/30/2020 12:15				3/30/2020 13:40		3/30/2020 13:40		3/30/2020 13:40			
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit																								
Soil pH																											
pH	N/A	N/A	N/A	7.0		7.0		7.2		1.63	7.36	5.5		6.1		6.3		6.98	7.01	6.8		7.0		6.9		1.45	7.15
Mercury																											
Mercury	7439-97-6	0.013	mg/kg	**0.25**	J	**0.15**	J	**0.15**	J	31.5	**0.329**	**0.26**		**0.26**		**0.21**		11.9	**0.316**	**0.28**		**0.31**		**0.36**		12.8	**0.418**
Target Analyte List Metals																											
Aluminum	7429-90-5	NS	mg/kg	9,700		11,000		10,000		7	11,900	11,000		11,000		11,000		0	11,000	11,000		11,000		12,000		5	12,800
Antimony	7440-36-0	0.27	mg/kg	**0.74**		**0.83**		**0.9**		9.74	**1.03**	**0.7**		**0.63**		**0.58**		9.47	**0.788**	**1.2**		**1.4**		**2.5**		41.2	**3.46**
Arsenic	7440-38-2	6.8	mg/kg	**37**		**43**		**39**		7.7	**47.4**	**13**		**14**		**14**		4.22	**15.1**	**9.7**		**11**		**11**		7.1	**12.5**
Barium	7440-39-3	110	mg/kg	76		79		78		1.97	81.5	84		79		79		3.58	87.9	88		86		96		5.88	103
Beryllium	7440-41-7	2.5	mg/kg	0.55		0.59		0.51		7.27	0.651	0.69		0.62		0.63		5.85	0.742	0.61		0.55		0.64		7.64	0.715
Cadmium	7440-43-9	0.36	mg/kg	**0.37**		0.36		**0.37**		1.57	**0.381**	**0.38**		0.33		0.35		7.12	**0.417**	**0.74**		**0.77**		**0.75**		2.03	**0.792**
Calcium	7440-70-2	NS	mg/kg	28,000		25,000		27,000		5.73	30,500	10,000		11,000		13,000		13.5	15,200	9,200		8,500		8,900		3.96	9,750
Chromium, Total	7440-47-3	23	mg/kg	15	J	15	J	15	J	0	15	15		15		15		0	15	15		14		15		3.94	16.1
Cobalt	7440-48-4	13	mg/kg	6.7		7		6.7		2.55	7.24	7.6		7.9		7.5		2.72	8.19	8.6		8.2		8.5		2.47	8.96
Copper	7440-50-8	28	mg/kg	**30**		**37**		**33**		10.5	**42.2**	**51**		**48**		**47**		4.28	**53.9**	**220**		**600**		**260**		58	**885**
Iron	7439-89-6	NS	mg/kg	17,000	J	19,000		19,000		6.3	21,200	17,000		17,000		17,000		0	17,000	18,000		18,000		19,000		3.15	19,800
Lead	7439-92-1	11	mg/kg	**130**		**110**		**130**		9.36	**152**	**82**		**83**		**89**		4.47	**94.2**	**190**		**150**		**160**		12.5	**219**
Magnesium	7439-95-4	NS	mg/kg	19,000		17,000		18,000		5.56	20,500	7,100		7,200		8,600		11	9,740	5,700		5,500		5,400		2.76	5,920
Manganese	7439-96-5	220	mg/kg	**600**		**600**		**600**		0	**600**	**940**		**820**		**790**		9.34	**1,050**	**1,000**		**830**		**1,100**		14	**1,320**
Nickel	7440-02-0	38	mg/kg	16		16		16		0	16	15		15		15		0	15	16		16		17		3.53	17.8
Potassium	2023695	NS	mg/kg	910		960		1,000		4.71	1,070	1,100		1,100		1,100		0	1,100	1,400		1,400		1,400		0	1,400
Selenium	7782-49-2	0.52	mg/kg	0.47	J	**0.61**		**0.57**		13.1	**0.731**	0.51		**0.54**		0.24	J	38.4	**0.846**	**0.55**		**0.6**		**0.68**		10.7	**0.775**
Silver	7440-22-4	4.2	mg/kg	0.073	J	0.082	J	0.084	J	7.35	0.0944	0.069	J	0.069	J	0.075	J	4.88	0.0797	0.086	J	0.094	J	0.089	J	4.51	0.0998
Sodium	7440-23-5	NS	mg/kg	61	J	56	J	64	J	6.7	70.5	73	J	67	J	68	J	4.64	77.4	120	J	120	J	130	J	4.68	138
Thallium	7440-28-0	0.05	mg/kg	**0.19**		**0.22**		**0.22**		8.25	**0.254**	**0.15**		**0.14**		**0.15**		3.94	**0.161**	**0.16**		**0.16**		**0.18**		6.93	**0.196**
Vanadium	7440-62-2	7.8	mg/kg	**22**	J	**24**	J	**23**	J	4.35	**25.5**	**28**		**26**		**27**		3.7	**29.5**	**30**		**30**		**31**		1.9	**31.8**
Zinc	7440-66-6	46	mg/kg	**87**		**100**		**110**		11.6	**128**	**95**		**100**		**93**		3.76	**105**	**140**		**150**		**170**		9.96	**192**
Polycyclic Aromatic Hydrocarbons																											
1-Methylnaphthalene	90-12-0	29	mg/kg	0.028	J	0.027	J	0.023	J	10.2	0.0327	0.073	J	0.044	J	0.053	J	26.2	0.094	0.38	J	0.45	J	0.26	J	26.4	0.605
2-Methylnaphthalene	91-57-6	29	mg/kg	0.043	J	0.044	J	0.036	J	10.6	0.052	0.098	J	0.055	J	0.053	J	37	0.133	0.43	J	0.5	J	0.31	J	23.2	0.655
Acenaphthene	83-32-9	29	mg/kg	0.02	J	0.029	J	0.015	J	33.3	0.0392	0.54	J	0.36	J	0.39	J	22.4	0.673	4.5	J	4.7	J	2.6	J	29.5	6.85
Acenaphthylene	208-96-8	29	mg/kg	0.17	J	0.23	J	0.16	J	20.3	0.282	0.088	J	0.029	J	0.062	J	49.6	0.134	0.21	UJ (LOD)	1.3	J	0.2	UJ (LOD)	111	2.16
Anthracene	120-12-7	29	mg/kg	0.13	J	0.15	J	0.097	J	21.3	0.193	0.86	J	0.59	J	0.75	J	18.5	1.08	6.6	J	8	J	3.9	J	33.8	11.4
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.67	J	0.97	J	0.52	J	31.8	**1.3**	**2.2**	J	**1.5**	J	**2.1**	J	19.6	**2.89**	**16**	J	**17**	J	**9.4**	J	29.2	**24.5**
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.87	J	**1.2**	J	0.75	J	24.8	**1.53**	**1.8**	J	**1.2**	J	**1.8**	J	21.7	**2.47**	**12**	J	**13**	J	**7.1**	J	29.5	**18.6**
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	**1.2**	J	**1.6**	J	1.1	J	20.4	**1.97**	**2.8**	J	**1.6**	J	**2.6**	J	27.6	**3.95**	**17**	J	**17**	J	**9.7**	J	28.9	**25.2**
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.82	J	**1.2**	J	0.78	J	24.8	**1.52**	**1.4**	J	0.69	J	**1.3**	J	34	**2.1**	**7.2**	J	**7.9**	J	**4.2**	J	30.6	**11.4**
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.43	J	0.57	J	0.36	J	23.6	0.722	1.1	J	0.62	J	0.97	J	27.7	**1.52**	**6.4**	J	**7.4**	J	**3.8**	J	31.7	**10.5**
Chrysene	218-01-9	1.1	mg/kg	0.96	J	**1.3**	J	0.75	J	27.7	**1.7**	**2.4**	J	**1.5**	J	**2.2**	J	23.2	**3.22**	**16**	J	**16**	J	**9.3**	J	28.1	**23.5**
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.18	J	0.23	J	0.16	J	19	0.281	0.38	J	0.2	J	0.37	J	31.9	0.571	**2.3**	J	**2.4**	J	**1.2**	J	33.9	**3.64**
Fluoranthene	206-44-0	1.1	mg/kg	1.1	J	**1.3**	J	0.71	J	28.9	**1.79**	**4.2**	J	**3.1**	J	**4.3**	J	17.2	**5.54**	**34**	J	**38**	J	**21**	J	28.7	**53.4**
Fluorene	86-73-7	29	mg/kg	0.043	J	0.061	J	0.028	J	37.6	0.0856	0.44	J	0.28	J	0.32	J	24	0.556	3.2	J	3.9	J	2	J	31.7	5.45
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.78	J	1.1	J	0.74	J	22.6	**1.37**	**1.5**	J	0.76	J	**1.4**	J	32.9	**2.23**	**7.9**	J	**9.2**	J	**4.9**	J	30.1	**12.9**
Naphthalene	91-20-3	29	mg/kg	0.098	J	0.092	J	0.068	J	18.5	0.126	0.25	J	0.12	J	0.086	J	56.9	0.37	0.95	J	1.1	J	0.75	J	18.8	1.38
Phenanthrene	85-01-8	29	mg/kg	0.42	J	0.42	J	0.22	J	32.7	0.644	3.2	J	2.3	J	2.7	J	16.5	3.87	26	J	27	J	15	J	29.4	**39.4**
Pyrene	129-00-0	1.1	mg/kg	**1.2**	J	**1.6**	J	0.87	J	29.9	**2.14**	**3.5**	J	**2.5**	J	**3.6**	J	19	**4.73**	**27**	J	**30**	J	**17**	J	27.6	**41.8**
Total LMW PAHs	N/A	29	mg/kg	1.622		2.023		1.167		26.7	2.68	7.749		5.278		6.514		19	9.62	**58.06**		**63.95**		**34.22**		30.2	**91.7**
Total HMW PAHs	N/A	1.1	mg/kg	**8.632**		**11.493**		**7.037**		24.9	**14.7**	**25.529**		**16.448**		**23.554**		21.9	**33.9**	**176.86**		**191.85**		**104.82**		29.5	**275**

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.

Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 4b DU-1 Through DU-16 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-14 (Previous Sampling Location II-EA-SS-02-13)					DU-15 (Previous Sampling Location II-EA-SS-02-15)					DU-16 (AOC #4: Former Building 124 to 128 – Sewage Disposal Plant [and Suspected Outfall])				
			Sample ID	IINY-DU-14A-SSIS-20200320	IINY-DU-14B-SSIS-20200320	IINY-DU-14C-SSIS-20200320	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-15A-SSIS-20200401	IINY-DU-15B-SSIS-20200401	IINY-DU-15C-SSIS-20200401	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-16A-SSIS-20200331	IINY-DU-16B-SSIS-20200331	IINY-DU-16C-SSIS-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in.)	0-6	0-6	0-6			0-6	0-6	0-6			0-6	0-6	0-6		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
			Sample Date	3/20/2020 8:30	3/20/2020 8:30	3/20/2020 8:30			4/1/2020 8:20	4/1/2020 8:20	4/1/2020 8:20			3/31/2020 9:50	3/31/2020 9:50	3/31/2020 9:50		
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit															
Soil pH																		
pH	N/A	N/A	N/A	6.7	6.9	7.0	2.22	7.25	6.2	6.3	6.6	3.27	6.89	6.9	6.9	7.4	4.09	7.79
Mercury																		
Mercury	7439-97-6	0.013	mg/kg	**0.16**	**0.18**	**0.19**	8.65	**0.215**	**0.14**	**0.21**	**0.29**	35.2	**0.402**	**0.097**	**0.096**	**0.096**	0.599	**0.0978**
Target Analyte List Metals																		
Aluminum	7429-90-5	NS	mg/kg	10,000	11,000	10,000	6	11,800	11,000	11,000	11,000	0	11,000	15,000	14,000	13,000	7	16,500
Antimony	7440-36-0	0.27	mg/kg	**0.6**	**0.56**	**0.59**	3.57	**0.636**	**1.1**	**0.7**	**0.7**	27.7	**1.41**	**0.44**	**0.4**	**0.42**	4.76	**0.47**
Arsenic	7440-38-2	6.8	mg/kg	**16**	**18**	**17**	5.88	**19.5**	**7**	6.5	**7.1**	4.68	**7.68**	**7.1**	6.8	**7.2**	2.96	**7.56**
Barium	7440-39-3	110	mg/kg	76	81	78	3.21	84.7	58	61	62	3.45	65.6	61	56	59	4.29	65
Beryllium	7440-41-7	2.5	mg/kg	0.53	0.57	0.55	3.64	0.6	0.47	0.5	0.47	3.61	0.524	0.6	0.6	0.53	7.01	0.678
Cadmium	7440-43-9	0.36	mg/kg	0.28	0.27	0.28	2.09	0.291	**0.58**	**0.38**	**0.58**	22.5	**0.804**	0.25	0.33	0.27	14.7	**0.388**
Calcium	7440-70-2	NS	mg/kg	9,500 J	8,900 J	6,800 J	16.9	12,000	7,100	7,500	9,900	18.5	12,000	8,000	7,600	7,200	5.26	8,610
Chromium, Total	7440-47-3	23	mg/kg	13	14	14	4.22	15.1	15	15	15	0	15	15	15	14	3.94	16.1
Cobalt	7440-48-4	13	mg/kg	6.8	6.9	7.3	3.78	7.67	6.8	6.7	6.8	0.853	6.91	8.5	8	**8.4**	3.19	8.97
Copper	7440-50-8	28	mg/kg	23	26	25	6.19	**28.5**	**30** J	**31** J	**33** J	4.88	**35.2**	**38**	**33**	**39**	8.77	**44.8**
Iron	7439-89-6	NS	mg/kg	16,000	17,000	16,000 J	3.53	17,800	19,000	18,000	18,000	3.15	19,800	22,000	20,000	21,000	4.76	23,500
Lead	7439-92-1	11	mg/kg	**96**	**120**	**110**	11.1	**139**	**130**	**130**	**120**	4.56	**141**	**38**	**42**	**46**	9.52	**52.1**
Magnesium	7439-95-4	NS	mg/kg	7,100 J	6,800 J	5,500 J	13.2	8,610	6,000	5,900	7,700	15.5	9,080	6,500	6,200	6,100	3.32	6,790
Manganese	7439-96-5	220	mg/kg	**670**	**720**	**700**	3.61	**760**	**410**	**480**	**430**	8.19	**531**	**450**	**430**	**440**	2.27	**465**
Nickel	7440-02-0	38	mg/kg	15	16	16	3.69	17.1	14	14	15	4.03	15.8	16	16	16	0	16
Potassium	2023695	NS	mg/kg	980	1,000	1,000	1.16	1,020	1,200	1,200	1,200	0	1,200	1,700	1,600	1,400	9.75	1,950
Selenium	7782-49-2	0.52	mg/kg	0.38 J	0.38 J	0.4 J	2.99	0.416	0.52	**0.62**	0.47 J	14.2	**0.729**	0.4 J	0.4 J	0.35 J	7.53	0.456
Silver	7440-22-4	4.2	mg/kg	0.08 J	0.063 J	0.087 J	16.1	0.108	0.079 J	0.089	0.073 J	10.1	0.101	0.055 J	0.063 J	0.057 J	7.14	0.0688
Sodium	7440-23-5	NS	mg/kg	72 J	70 J	73 J	2.13	75.5	75 J	81 J	77 J	3.93	85.4	440 J	360 J	330 J	15.1	520
Thallium	7440-28-0	0.05	mg/kg	**0.11**	**0.13**	**0.12**	8.33	**0.145**	**0.16**	**0.15**	**0.16**	3.69	**0.171**	**0.13**	**0.12**	**0.12**	4.68	**0.138**
Vanadium	7440-62-2	7.8	mg/kg	**20**	**20**	**20**	0	**20**	**25**	**26**	**25**	2.28	**26.8**	**28**	**30**	**30**	3.94	**32.2**
Zinc	7440-66-6	46	mg/kg	**74**	**74**	**78**	3.07	**81.1**	**250**	**120**	**120**	46	**352**	**70**	**71**	**66**	3.83	**75.7**
Polycyclic Aromatic Hydrocarbons																		
1-Methylnaphthalene	90-12-0	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.026 J	0.028	0.0077 J	54.4	0.0487
2-Methylnaphthalene	91-57-6	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.033 J	0.041	0.0082 J	62.4	0.0704
Acenaphthene	83-32-9	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.15 J	0.14	0.036	58.1	0.268
Acenaphthylene	208-96-8	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.032 J	0.034	0.028	9.75	0.039
Anthracene	120-12-7	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.18 J	0.16	0.063	46.6	0.292
Benzo(a)anthracene	56-55-3	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.5 J	0.44	0.21	39.9	0.769
Benzo(a)pyrene	50-32-8	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.47 J	0.43	0.24	32.3	0.689
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.71 J	0.65	0.33	36.3	1.08
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.31 J	0.29	0.15	34.9	0.469
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.26 J	0.23	0.12	36.3	0.389
Chrysene	218-01-9	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.49 J	0.44	0.21	39.3	0.756
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.1 J	0.085	0.042	39.8	0.151
Fluoranthene	206-44-0	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	1 J	0.96	0.41	41.7	**1.62**
Fluorene	86-73-7	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.1 J	0.1	0.027	55.7	0.182
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.38 J	0.35	0.19	33.3	0.564
Naphthalene	91-20-3	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.064 J	0.089	0.012	71.4	0.154
Phenanthrene	85-01-8	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.74 J	0.66	0.2	54.6	1.27
Pyrene	129-00-0	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.86 J	0.8	0.35	41.6	**1.37**
Total LMW PAHs	N/A	29	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	1.825	1.692	0.5919	49.4	3.07
Total HMW PAHs	N/A	1.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	**6.045**	**5.567**	**2.4939**	41	**9.55**

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-8 (AOC #5: Former Building 207 – Marine Garage Building) was not sampled because it would not be possible to separate metals and PAH contaminants originating from former Building 207, from contaminants originating from drums and other debris observed in DU-8.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 5a DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-17 (AOC #2 - Former Building 121 [Incinerator], AOC #16 - Area D - Dumping Area, and Previous Sampling Locations IX-121-B1 and II-EA-SD-02-02										
				Sample ID	IINY-DU-17A-SSIS-20200331	IINY-DU-17B-SSIS-20200331	IINY-DU-17C-SSIS-20200331	%RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-17A-SBIS1-20200331	IINY-DU-17B-SBIS1-20200331	IINY-DU-17C-SBIS1-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
				Sample Date	3/31/2020 8:15	3/31/2020 8:15	3/31/2020 8:15			3/31/2020 8:45	3/31/2020 8:45	3/31/2020 8:45		
Analyte	CAS #	Residential Soil RSL (mg/kg)[ab]	Industrial Soil RSL (mg/kg)[a]	Unit										
Soil pH														
pH	N/A	N/A	N/A	N/A	7.2	7.2	7.1	0.806	7.31	7.0	7.4	7.7	4.77	8.25
Mercury														
Mercury	7439-97-6	1.1	4.6	mg/kg	0.98	0.61	0.77	24	1.25	0.9	0.8	0.77	8	0.995
Target Analyte List Metals														
Aluminum	7429-90-5	7,700	110,000	mg/kg	5,400	5,300	7,000	16	8,300	6,100	5,900	6,100	2	6,320
Antimony	7440-36-0	3.1	47	mg/kg	9.9	7.3	6.5	22.5	12.4	4.8	4.5	4.4	4.56	5.09
Arsenic	7440-38-2	0.68	3	mg/kg	8.2	2.7	8.8	51.2	15	22	25	23	6.55	27.2
Barium	7440-39-3	1,500	22,000	mg/kg	150	120	130	11.5	172	460	330	280	26.1	590
Beryllium	7440-41-7	16	230	mg/kg	0.38	0.15	0.53	54.2	0.835	1.2	1.3	1.5	11.5	1.72
Cadmium	7440-43-9	7.1	98	mg/kg	5	20	16	56.8	33.2	3.9	3.4	3.2	10.3	4.41
Calcium	7440-70-2	NS	NS	mg/kg	74,000	43,000	58,000	26.6	97300	14,000	19,000	22,000	22	28,500
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	19	6.9	25	54.3	40.2	13	13	12	4.56	14.1
Cobalt	7440-48-4	2.3	35	mg/kg	7.2	4.9	5.7	19.7	8.87	6.3	6.8	6.6	3.83	7.2
Copper	7440-50-8	310	4,700	mg/kg	210	83	280	52.3	442	180	130	130	19.7	219
Iron	7439-89-6	5,500	82,000	mg/kg	16,000	14,000	19,000	15.4	22,700	26,000	26,000	25,000	2.25	27,100
Lead	7439-92-1	400	800	mg/kg	2,900	1,500	1,200	48.6	4,150	2,700	1,200	820	63.2	4,070
Magnesium	7439-95-4	NS	NS	mg/kg	45,000	26,000	34,000	27.3	59,000	6,400	8,500	9,800	20.8	12,600
Manganese	7439-96-5	180	2,600	mg/kg	400	380	440	7.51	484	170	170	170	0	170
Nickel	7440-02-0	150	2,200	mg/kg	16	5.9	20	52	32.3	19	21	19	5.87	22.6
Potassium	2023695	NS	NS	mg/kg	1100	1100	1400	14.4	1640	610	550	560	5.61	654
Selenium	7782-49-2	39	580	mg/kg	0.52	0.2 J	0.68	52.4	1.08	1.1	1.4	1.5	15.6	1.86
Silver	7440-22-4	39	580	mg/kg	0.59	0.56	0.72	13.6	0.837	0.3	0.25	0.24	12.2	0.344
Sodium	7440-23-5	NS	NS	mg/kg	400 J	370 J	490	14.9	577	510	480	510	3.46	544
Thallium	7440-28-0	0.078	1.2	mg/kg	0.12	0.1	0.15	20.4	0.187	0.6	0.72	0.8	14.2	0.96
Vanadium	7440-62-2	39	580	mg/kg	17	18	22	13.9	25.7	24	22	21	6.84	26.2
Zinc	7440-66-6	2,300	35,000	mg/kg	8,200	7,200	5,700	17.9	10,200	1,900	2,900	1,600	31.9	3,850
Polycyclic Aromatic Hydrocarbons														
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.03	0.026	0.037	18	0.045	0.087 J	0.098 J	0.11 J	11.7	0.127
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.044	0.039	0.056	18.900	0.0683	0.12 J	0.14 J	0.16 J	14.300	0.19
Acenaphthene	83-32-9	360	4,500	mg/kg	0.035	0.028	0.044	22.500	0.0559	0.068 J	0.058 J	0.087 J	20.700	0.108
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.13	0.11	0.18	25.800	0.231	0.031 J	0.035 J	0.042 J	15.500	0.05
Anthracene	120-12-7	1,800	23,000	mg/kg	0.17	0.15	0.24	25.300	0.306	0.21	0.081 J	0.16 J	43.300	0.314
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.58	0.48 J	0.89	32.900	1.19	0.7	0.2 J	0.47 J	54.800	1.09
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.5	0.41 J	0.75	31.800	0.997	0.45	0.11 J	0.31 J	58.900	0.72
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	0.88	0.71 J	1.3	31.500	1.73	0.97	0.32 J	0.67 J	49.800	1.47
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.32	0.29	0.45	24.100	0.567	0.34	0.13 J	0.27 J	43.300	0.516
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.28	0.24	0.38	24.000	0.481	0.23 J	0.051 J	0.17 J	60.600	0.38
Chrysene	218-01-9	110	2,100	mg/kg	0.66	0.57 J	1	30.500	1.31	1	0.48 J	0.8 J	34.500	1.42
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.12	0.093	0.15	23.600	0.193	0.15 J	0.068 J	0.13 J	36.900	0.224
Fluoranthene	206-44-0	240	3,000	mg/kg	1.1	0.94 J	1.6	28.400	2.08	1.5	0.44 J	1 J	54.100	2.31
Fluorene	86-73-7	240	3,000	mg/kg	0.043	0.035	0.06	27.800	0.0781	0.076 J	0.064 J	0.11 J	28.600	0.143
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.38	0.3 J	0.52	27.800	0.68	0.29	0.085 J	0.25 J	52.200	0.482
Naphthalene	91-20-3	2	8.6	mg/kg	0.073	0.069	0.094	17.100	0.112	0.2 J	0.24 J	0.28 J	16.700	0.341
Phenanthrene	85-01-8	NS	NS	mg/kg	0.49	0.4 J	0.74	32.400	0.987	1.1	0.48 J	0.88 J	38.300	1.61
Pyrene	129-00-0	180	2,300	mg/kg	1.1	0.92 J	1.6	29.200	2.09	1.2	0.4 J	0.87 J	48.800	1.84
Total LMW PAHs	N/A	NS	NS	mg/kg	1.015	0.857	1.451	27.800	1.88	1.892	1.196	1.829	23.500	2.61
Total HMW PAHs	N/A	NS	NS	mg/kg	5.92	4.953	8.64	29.400	11.3	6.83	2.284	4.94	48.700	10.4
Polychlorinated Biphenyls														
PCB, Total	1336-36-3	0.23	0.94	mg/kg	0.14	0.13	0.14	4.22	0.151	0.016 UJ (LOD)	0.015 UJ (LOD)	0.015 UJ (LOD)	ND	ND
PCB-1016 (Aroclor 1016)	12674-11-2	0.41	5.1	mg/kg	0.03 U (LOD)	0.027 U (LOD)	0.027 U (LOD)	ND	ND	0.031 UJ (LOD)	0.029 UJ (LOD)	0.028 UJ (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.2	0.83	mg/kg	0.078 U (LOD)	0.071 U (LOD)	0.071 U (LOD)	ND	ND	0.082 UJ (LOD)	0.075 UJ (LOD)	0.073 UJ (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.17	0.72	mg/kg	0.03 U (LOD)	0.027 U (LOD)	0.027 U (LOD)	ND	ND	0.031 UJ (LOD)	0.029 UJ (LOD)	0.028 UJ (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.23	0.95	mg/kg	0.047 U (LOD)	0.043 U (LOD)	0.043 U (LOD)	ND	ND	0.049 UJ (LOD)	0.045 UJ (LOD)	0.044 UJ (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.23	0.95	mg/kg	0.095	0.084	0.087	6.41	0.103	0.015 UJ (LOD)	0.014 UJ (LOD)	0.014 UJ (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.12	0.97	mg/kg	0.032 U (LOD)	0.03 U (LOD)	0.03 U (LOD)	ND	ND	0.034 UJ (LOD)	0.031 UJ (LOD)	0.03 UJ (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.24	0.99	mg/kg	0.044 J	0.041 J	0.048 J	7.92	0.0532	0.0068 UJ (LOD)	0.0063 UJ (LOD)	0.0031 J	37.2	0.0105
PCB-1262 (Aroclor 1262)	37324-23-5	NS	NS	mg/kg	0.016 U (LOD)	0.014 U (LOD)	0.014 U (LOD)	ND	ND	0.016 UJ (LOD)	0.015 UJ (LOD)	0.015 UJ (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	NS	NS	mg/kg	0.0065 UJ (LOD)	0.0059 UJ (LOD)	0.0059 UJ (LOD)	ND	ND	0.0068 UJ (LOD)	0.0063 UJ (LOD)	0.0061 UJ (LOD)	ND	ND
Explosives Residues														
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.038 U (LOD)	0.037 UJ (LOD)	0.037 U (LOD)	ND	ND	0.038 U (LOD)	0.038 U (LOD)	0.038 U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.096 UJ (LOQ)	0.093 UJ (LOQ)	0.092 UJ (LOQ)	ND	ND	0.095 UJ (LOQ)	0.095 UJ (LOQ)	0.096 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.096 U (LOQ)	0.093 UJ (LOQ)	0.092 U (LOQ)	ND	ND	0.095 U (LOQ)	0.095 U (LOQ)	0.096 UJ (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.096 U (LOD)	0.093 UJ (LOD)	0.092 U (LOD)	ND	ND	0.095 U (LOD)	0.095 U (LOD)	0.096 U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.019 U (LOD)	0.019 UJ (LOD)	0.018 U (LOD)	ND	ND	0.019 U (LOD)	0.019 U (LOD)	0.019 U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.18 UJ (LOQ)	ND	ND	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.096 U (LOQ)	0.093 UJ (LOQ)	0.092 U (LOQ)	ND	ND	0.095 U (LOQ)	0.095 U (LOQ)	0.096 U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.096 UJ (LOD)	0.093 UJ (LOD)	0.092 UJ (LOD)	ND	ND	0.095 UJ (LOD)	0.095 UJ (LOD)	0.096 UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.096 U (LOD)	0.093 U (LOD)	0.092 U (LOD)	ND	ND	0.095 U (LOD)	0.095 U (LOD)	0.096 U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.19 UJ (LOD)	0.19 UJ (LOD)	0.18 UJ (LOD)	ND	ND	0.19 UJ (LOD)	0.19 UJ (LOD)	0.19 UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.67 U (LOD)	0.65 UJ (LOD)	0.64 U (LOD)	ND	ND	0.67 U (LOD)	0.67 U (LOD)	0.67 U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	390	5,700	mg/kg	0.067 U (LOD)	0.065 UJ (LOD)	0.064 U (LOD)	ND	ND	0.067 U (LOD)	0.067 U (LOD)	0.067 U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.96 U (LOD)	0.93 UJ (LOD)	0.92 U (LOD)	ND	ND	0.95 U (LOD)	0.95 U (LOD)	0.96 U (LOD)	ND	ND
Tetryl	479-45-8	0.16	230	mg/kg	0.19 U (LOQ)	0.093 UJ (LOD)	0.092 UJ (LOD)	ND	ND	0.095 UJ (LOD)	0.095 UJ (LOD)	0.096 UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 5a DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-18 (AOC #16 - Area D - Dumping Area and Previous Sampling Locations IX DMP-B22 and IXDMP-B1)											
				Sample ID	IINY-DU-18A-SSIS-20200331	IINY-DU-18B-SSIS-20200331	IINY-DU-18C-SSIS-20200331	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-18A-SBIS1-20200331	IINY-DU-18B-SBIS1-20200331	IINY-DU-18B-SBIS1-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
				Sample Date	3/31/2020 13:45	3/31/2020 13:45	3/31/2020 13:45			3/31/2020 14:20	3/31/2020 14:20	3/31/2020 14:20		
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit										
Soil pH														
pH	N/A	N/A	N/A	N/A	7.5	8.0	7.8	3.24	8.4	8.1	8.1	8	0.716	0
Mercury														
Mercury	7439-97-6	1.1	4.6	mg/kg	4.3	2.3	1.5	53	6.33	0.71	2.1	0.71	68	3.19
Target Analyte List Metals														
Aluminum	7429-90-5	7,700	110,000	mg/kg	11,000	11,000	6,500	27	16,000	4,300	4,800	5,900	16	7,060
Antimony	7440-36-0	3.1	47	mg/kg	6.5	6.2	6.9	5.38	7.42	11	13	5.5	39.5	19.6
Arsenic	7440-38-2	0.68	3	mg/kg	7.8	15	15	33	23.1	17	19	16	8.81	21.2
Barium	7440-39-3	1,500	22,000	mg/kg	360	230	240	26.1	459	330	310	270	10.1	380
Beryllium	7440-41-7	16	230	mg/kg	0.56	0.55	0.66	10.3	0.743	0.64	0.69	0.63	4.92	0.734
Cadmium	7440-43-9	7.1	98	mg/kg	5.2	4.2	4.5	11.1	5.92	7.6	24	4.7	86	38.3
Calcium	7440-70-2	NS	NS	mg/kg	12,000	12,000	13,000	4.68	13,800	44,000	32,000	20,000	37.5	62,200
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	14	33	240	131	411	33	32	29	6.64	36.6
Cobalt	7440-48-4	2.3	35	mg/kg	7.6	7	7.2	4.2	8.04	7.5	5	6.5	19.9	9.5
Copper	7440-50-8	310	4,700	mg/kg	40	1,700	1,000	91.2	3,010	5,500	5,600	5,100	4.9	6,070
Iron	7439-89-6	5,500	82,000	mg/kg	35,000	35,000	27,000	14.3	44,000	43000	36000	33000	13.7	50,200
Lead	7439-92-1	400	800	mg/kg	1,500	1,400	1,400	4.03	1,580	2,900	2,000	2,400	18.5	3,570
Magnesium	7439-95-4	NS	NS	mg/kg	7900	7800	7,900	0.734	8,010	24,000	17,000	11,000	37.5	33,700
Manganese	7439-96-5	180	2,600	mg/kg	540	550	450	10.7	652	480	450	510	6.25	555
Nickel	7440-02-0	150	2,200	mg/kg	16	29	30	31.2	44.7	79	64	52	20.8	99
Potassium	2023695	NS	NS	mg/kg	2,300	2,300	920	43.3	3,850	480	590	1100	45.7	1560
Selenium	7782-49-2	39	580	mg/kg	0.36 J	0.74	0.58	34.1	1.04	0.79	0.95	0.86	9.25	1.07
Silver	7440-22-4	39	580	mg/kg	0.92	0.63	0.85	18.9	1.18	1.5	2.1	0.83	43	3.08
Sodium	7440-23-5	NS	NS	mg/kg	810	810	590	17.2	1060	420 J	430 J	430 J	1.35	441
Thallium	7440-28-0	0.078	1.2	mg/kg	0.13	0.21	0.2	24.2	0.29	0.35	0.31	0.27	12.9	0.411
Vanadium	7440-62-2	39	580	mg/kg	29	24	29 J	10.6	34.6	17	18	18	3.27	19.1
Zinc	7440-66-6	2,300	35,000	mg/kg	83	1,800	1,600	80.9	3,520	5,600	5,000	3,100	28.6	7,850
Polycyclic Aromatic Hydrocarbons														
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.04	0.04 J	0.041 J	1.43	0.0418	0.1 J	0.08 J	0.086 J	11.6	0.114
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.07	0.06 J	0.072 J	9.550	0.0835	0.12 J	0.095 J	0.1 J	12.600	0.138
Acenaphthene	83-32-9	360	4,500	mg/kg	0.046	0.055 J	0.052 J	8.990	0.0625	0.23 J	0.18 J	0.18 J	14.700	0.269
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.059	0.074 J	0.12 J	37.700	0.164	0.041 J	0.063 J	0.066 J	24.100	0.091
Anthracene	120-12-7	1,800	23,000	mg/kg	0.13	0.17 J	0.21 J	23.500	0.271	0.12 J	0.16 J	0.23 J	32.800	0.31
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.49 J	0.63 J	1	37.300	1.37	0.28 J	0.44 J	0.79	51.800	1.16
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.44 J	0.58 J	0.92	38.200	1.27	0.15 J	0.28 J	0.64	71.200	0.995
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	0.86 J	1.1 J	1.6	31.800	2.14	0.44 J	0.77 J	1.3	51.900	1.93
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.3	0.41 J	0.64	38.600	0.887	0.14 J	0.24 J	0.51	64.500	0.778
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.21	0.31 J	0.45	37.300	0.627	0.075 J	0.14 J	0.28	63.500	0.429
Chrysene	218-01-9	110	2,100	mg/kg	0.86 J	1.1 J	1.5	28.000	1.97	0.65 J	1.1 J	1.7	45.800	2.48
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.11	0.16 J	0.19 J	26.400	0.255	0.064 J	0.1 J	0.2 J	58.100	0.299
Fluoranthene	206-44-0	240	3,000	mg/kg	0.97 J	1.2 J	1.7	28.900	2.23	0.72 J	0.93 J	1.4	34.200	1.89
Fluorene	86-73-7	240	3,000	mg/kg	0.061	0.071 J	0.068 J	7.700	0.0796	0.15 J	0.14 J	0.16 J	6.670	0.175
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.31	0.45 J	0.71	41.400	1	0.1 J	0.22 J	0.45	69.300	0.704
Naphthalene	91-20-3	2	8.6	mg/kg	0.24	0.2 J	0.24 J	10.200	0.285	0.71 J	0.46 J	0.51	23.600	0.893
Phenanthrene	85-01-8	NS	NS	mg/kg	0.62 J	0.78 J	0.9	18.300	1.12	0.66 J	0.86 J	1.4	39.300	1.94
Pyrene	129-00-0	180	2,300	mg/kg	0.87 J	1.1 J	1.7	35.000	2.3	0.62 J	0.89 J	1.3	36.600	1.8
Total LMW PAHs	N/A	NS	NS	mg/kg	1.266	1.45	1.703	14.900	2.03	2.131	2.038	2.732	16.400	3.25
Total HMW PAHs	N/A	NS	NS	mg/kg	5.42	7.04	10.41	33.400	14	3.239	5.11	8.57	48.000	12.4
Polychlorinated Biphenyls														
PCB, Total	1336-36-3	0.23	0.94	mg/kg	0.29	0.17	0.4 J	40.1	0.576	0.15	0.1	0.057 J	45.5	0.219
PCB-1016 (Aroclor 1016)	12674-11-2	0.41	5.1	mg/kg	0.029 U (LOD)	0.028 U (LOD)	0.029 UJ (LOD)	ND	ND	0.03 U (LOD)	0.028 U (LOD)	0.029 U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.2	0.83	mg/kg	0.075 U (LOD)	0.074 U (LOD)	0.076 UJ (LOD)	ND	ND	0.078 U (LOD)	0.072 U (LOD)	0.075 U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.17	0.72	mg/kg	0.029 U (LOD)	0.028 U (LOD)	0.029 UJ (LOD)	ND	ND	0.03 U (LOD)	0.028 U (LOD)	0.029 U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.23	0.95	mg/kg	0.045 U (LOD)	0.044 U (LOD)	0.046 UJ (LOD)	ND	ND	0.047 U (LOD)	0.043 U (LOD)	0.045 U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.23	0.95	mg/kg	0.13	0.099	0.14 J	17.4	0.177	0.015 U (LOD)	0.013 U (LOD)	0.014 U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.12	0.97	mg/kg	0.031 U (LOD)	0.031 U (LOD)	0.032 UJ (LOD)	ND	ND	0.032 U (LOD)	0.03 U (LOD)	0.031 U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.24	0.99	mg/kg	0.16	0.074	0.25 J	54.6	0.383	0.0065 U (LOD)	0.006 U (LOD)	0.0062 U (LOD)	ND	ND
PCB-1262 (Aroclor 1262)	37324-23-5	NS	NS	mg/kg	0.015 U (LOD)	0.015 U (LOD)	0.015 UJ (LOD)	ND	ND	0.15 J	0.1 J	0.057 J	45.5	0.219
PCB-1268 (Aroclor 1268)	11100-14-4	NS	NS	mg/kg	0.0063 UJ (LOD)	0.0061 UJ (LOD)	0.0063 UJ (LOD)	ND	ND	0.0065 U (LOD)	0.006 U (LOD)	0.0062 U (LOD)	ND	ND
Explosives Residues														
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.037 U (LOD)	0.039 U (LOD)	0.038 U (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.094 UJ (LOQ)	0.098 UJ (LOQ)	0.096 UJ (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.094 UJ (LOQ)	0.098 U (LOQ)	0.096 U (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.094 U (LOD)	0.098 U (LOD)	0.096 U (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.019 U (LOD)	0.02 U (LOD)	0.019 U (LOD)	ND	ND	0.019 UJ (LOD)	0.019 UJ (LOD)	0.019 UJ (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.19 UJ (LOQ)	0.2 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.094 U (LOQ)	0.098 U (LOQ)	0.096 U (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.094 UJ (LOQ)	0.098 UJ (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.094 UJ (LOD)	0.098 U (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.19 UJ (LOD)	0.2 UJ (LOD)	0.19 UJ (LOD)	ND	ND	0.19 UJ (LOD)	0.19 UJ (LOD)	0.19 UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.65 U (LOD)	0.68 U (LOD)	0.67 U (LOD)	ND	ND	0.68 UJ (LOD)	0.67 UJ (LOD)	0.66 UJ (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	390	5,700	mg/kg	0.065 U (LOD)	0.068 U (LOD)	0.067 U (LOD)	ND	ND	0.068 UJ (LOD)	0.067 UJ (LOD)	0.066 UJ (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.94 U (LOD)	0.98 U (LOD)	0.96 U (LOD)	ND	ND	0.96 UJ (LOD)	0.96 UJ (LOD)	0.94 UJ (LOD)	ND	ND
Tetryl	479-45-8	0.16	230	mg/kg	0.094 UJ (LOD)	0.098 UJ (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 5a DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-19 (AOC #16 - Area D - Dumping Area and Previous Sampling Location II-EA-SS-02-16													
				Sample ID	IINY-DU-19A-SSIS-20200331		IINY-DU-19B-SSIS-20200331		IINY-DU-19C-SSIS-20200331		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-19A-SBIS1-20200331		IINY-DU-19B-SBIS1-20200331		IINY-DU-19B-SBIS1-20200331		% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6		0-6		0-6				6-36		6-36		6-36			
				Matrix	Surface Soil		Surface Soil		Surface Soil				Subsurface Soil		Subsurface Soil		Subsurface Soil			
				Sample Date	3/31/2020 10:45		3/31/2020 10:45		3/31/2020 10:45				3/31/2020 11:00		3/31/2020 11:00		3/31/2020 11:00			
Analyte	CAS #	Residential Soil RSL (mg/kg)[ab]	Industrial Soil RSL (mg/kg)[a]	Unit																
Soil pH																				
pH	N/A	N/A	N/A	N/A	7.7		7.5		7.4		2.03	7.92	0.120		0.230		0.270		38	0.402
Mercury																				
Mercury	7439-97-6	1.1	4.6	mg/kg	0.46	J	0.11	J	0.43	J	58	0.822	0.12		0.23		0.27		38	0.402
Target Analyte List Metals																				
Aluminum	7429-90-5	7,700	110,000	mg/kg	16,000		25,000		20,000		22	31,700	25,000		20,000		21,000		12	28,700
Antimony	7440-36-0	3.1	47	mg/kg	0.5	J	0.28	J	0.5	J	29.8	0.746	0.12	J	0.11	J	0.11	J	5.09	0.128
Arsenic	7440-38-2	0.68	3	mg/kg	9.6	J	6.8	J	12	J	27.5	16	2.8		2.9		2.6		5.52	3.13
Barium	7440-39-3	1,500	22,000	mg/kg	81	J	63	J	83	J	14.6	103	34	J	37		35		4.32	39.2
Beryllium	7440-41-7	16	230	mg/kg	0.42	J	0.35	J	0.45	J	12.6	0.536	0.19		0.19		0.17		6.3	0.212
Cadmium	7440-43-9	7.1	98	mg/kg	0.33		0.25		0.3		13.8	0.395	0.097	J	0.093	J	0.087	J	5.45	0.105
Calcium	7440-70-2	NS	NS	mg/kg	10,000	J	15,000		12,000		20	18,700	20,000		17,000		17,000		10	22,400
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	10	J	10	J	11	J	5.59	11.8	7.8		7.7		7.8		0.743	7.91
Cobalt	7440-48-4	2.3	35	mg/kg	14		19		15		16.5	22.7	18		16		17		5.88	19.5
Copper	7440-50-8	310	4,700	mg/kg	130		180		140		17.6	217	210		210		210		0	210
Iron	7439-89-6	5,500	82,000	mg/kg	23,000	J	36,000		29,000		22	45,700	36,000		33,000		35,000		4	38,500
Lead	7439-92-1	400	800	mg/kg	61		38		67		27.7	93.9	14		12		11		12.4	16.2
Magnesium	7439-95-4	NS	NS	mg/kg	5,700	J	8,300	J	6,700	J	19	10,200	7,600		7,200		7,000		4	8,040
Manganese	7439-96-5	180	2,600	mg/kg	410	J	410	J	470	J	8.06	517	270	J	280		280		2.09	291
Nickel	7440-02-0	150	2,200	mg/kg	16	J	17	J	17	J	3.46	18.1	14		14		14		0	14
Potassium	2023695	NS	NS	mg/kg	1,200		1,500		1,600		15	1,960	1,200		1,100		1,100		5	1,280
Selenium	7782-49-2	39	580	mg/kg	0.42	J	0.35	J	0.44	J	11.7	0.522	0.23	J	0.23	J	0.23	J	0	0.23
Silver	7440-22-4	39	580	mg/kg	0.12		0.12		0.13		4.68	0.138	0.11		0.097	J	0.1		6.65	0.119
Sodium	7440-23-5	NS	NS	mg/kg	1,700		3,100		2,100		31	4,110	3,400		2,400		2,400		21	4,190
Thallium	7440-28-0	0.078	1.2	mg/kg	0.12	J	0.12	J	0.14	J	9.12	0.156	0.07	J	0.076	J	0.071	J	4.44	0.0804
Vanadium	7440-62-2	39	580	mg/kg	46		52		44		8.8	57.8	59		55		52		6.35	64.2
Zinc	7440-66-6	2,300	35,000	mg/kg	120	J	90	J	110	J	14.3	145	60		61		62		1.64	63.5
Polycyclic Aromatic Hydrocarbons																				
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.023		0.012		0.017		31.8	0.0312	0.0011	J	0.0012	J	0.00064	J	30.5	0.00173
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.035	J	0.018	J	0.028	J	31.600	0.0485	0.0017	J	0.0019	J	0.00093	J	33.900	0.0028
Acenaphthene	83-32-9	360	4,500	mg/kg	0.06		0.026		0.027		51.400	0.0864	0.0024	J	0.0015	J	0.00087	J	48.400	0.00353
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.27	J	0.13	J	0.21	J	34.500	0.38	0.0066	J	0.0069	J	0.0034	J	34.400	0.0105
Anthracene	120-12-7	1,800	23,000	mg/kg	0.43		0.19		0.24		44.200	0.605	0.0067	J	0.0064	J	0.0032	J	35.700	0.0103
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	2.5		1.1		1.1		51.600	3.6	0.024		0.029		0.013		37.200	0.0426
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	1.9		0.9		0.98		44.100	2.66	0.029		0.031		0.015		34.900	0.0469
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	3.4		1.6		1.8		43.500	4.75	0.045		0.047		0.023		34.700	0.0718
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	1.1		0.55		0.64		38.600	1.51	0.025		0.028		0.015		30.000	0.0398
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	1.2		0.59		0.59		44.400	1.68	0.013		0.015		0.0072	J	34.500	0.0219
Chrysene	218-01-9	110	2,100	mg/kg	2.3		0.98		1.1		50.000	3.3	0.027		0.033		0.014		39.400	0.0491
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.41		0.26		0.27		26.800	0.524	0.0061	J	0.0067	J	0.0039	J	26.500	0.00928
Fluoranthene	206-44-0	240	3,000	mg/kg	4		1.6		1.7		55.800	5.85	0.046		0.053		0.023		38.600	0.0802
Fluorene	86-73-7	240	3,000	mg/kg	0.077	J	0.028	J	0.039	J	53.600	0.113	0.0037	J	0.0028	J	0.0011	J	52.100	0.00586
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	1.5		0.77		0.82		39.600	2.06	0.027		0.029		0.014		34.900	0.0438
Naphthalene	91-20-3	2	8.6	mg/kg	0.083	J	0.046	J	0.068	J	28.300	0.113	0.0019	J	0.0022	J	0.0019	U (LOD)	8.660	0.00244
Phenanthrene	85-01-8	NS	NS	mg/kg	1.5		0.52		0.58		63.400	2.25	0.022		0.023		0.0091	J	43.000	0.0375
Pyrene	129-00-0	180	2,300	mg/kg	3.2		1.3		1.4		54.400	4.66	0.044		0.052		0.023		37.800	0.0774
Total LMW PAHs	N/A	NS	NS	mg/kg	2.478		0.97		1.209		52.200	3.59	0.0461		0.0459		0.02114		38.100	0.0738
Total HMW PAHs	N/A	NS	NS	mg/kg	21.51		9.65		10.4		47.900	30.6	0.2861		0.3237		0.1511		35.800	0.482
Polychlorinated Biphenyls																				
PCB, Total	1336-36-3	0.23	0.94	mg/kg	0.011	J	0.0071	J	0.015	UJ (LOD)	35.8	0.021	0.05	J	0.049	J	0.054	J	5.19	0.0577
PCB-1016 (Aroclor 1016)	12674-11-2	0.41	5.1	mg/kg	0.029	U (LOD)	0.028	U (LOD)	0.029	UJ (LOD)	ND	ND	0.03	U (LOD)	0.029	U (LOD)	0.03	U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.2	0.83	mg/kg	0.076	U (LOD)	0.072	U (LOD)	0.075	UJ (LOD)	ND	ND	0.077	U (LOD)	0.076	U (LOD)	0.077	U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.17	0.72	mg/kg	0.029	U (LOD)	0.028	U (LOD)	0.029	UJ (LOD)	ND	ND	0.03	U (LOD)	0.029	U (LOD)	0.03	U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.23	0.95	mg/kg	0.046	U (LOD)	0.043	U (LOD)	0.045	UJ (LOD)	ND	ND	0.046	U (LOD)	0.046	U (LOD)	0.046	U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.23	0.95	mg/kg	0.014	U (LOD)	0.013	U (LOD)	0.014	UJ (LOD)	ND	ND	0.015	U (LOD)	0.014	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.12	0.97	mg/kg	0.032	U (LOD)	0.03	U (LOD)	0.031	UJ (LOD)	ND	ND	0.032	U (LOD)	0.032	U (LOD)	0.032	U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.24	0.99	mg/kg	0.011	J	0.0071	J	0.0022	J	65.2	0.0179	0.05	J	0.049	J	0.054	J	5.19	0.0577
PCB-1262 (Aroclor 1262)	37324-23-5	NS	NS	mg/kg	0.015	U (LOD)	0.014	U (LOD)	0.015	UJ (LOD)	ND	ND	0.015	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	NS	NS	mg/kg	0.0063	U (LOD)	0.006	U (LOD)	0.0063	UJ (LOD)	ND	ND	0.0065	U (LOD)	0.0064	U (LOD)	0.0065	U (LOD)	ND	ND
Explosives Residues																				
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.038	U (LOD)	0.04	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.099	UJ (LOQ)	0.1	UJ (LOQ)	0.098	UJ (LOQ)	ND	ND	0.096	UJ (LOQ)	0.096	UJ (LOQ)	0.099	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.099	U (LOQ)	0.1	U (LOQ)	0.098	U (LOQ)	ND	ND	0.096	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.099	U (LOD)	0.1	U (LOD)	0.098	U (LOD)	ND	ND	0.096	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.02	UJ (LOD)	0.02	UJ (LOD)	0.02	UJ (LOD)	ND	ND	0.019	U (LOD)	0.019	U (LOD)	0.02	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.2	UJ (LOQ)	0.2	UJ (LOQ)	0.2	UJ (LOQ)	ND	ND	0.19	UJ (LOQ)	0.19	UJ (LOQ)	0.2	UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.099	U (LOQ)	0.1	U (LOQ)	0.098	U (LOQ)	ND	ND	0.096	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.099	UJ (LOD)	0.1	UJ (LOD)	0.098	UJ (LOD)	ND	ND	0.096	UJ (LOD)	0.096	UJ (LOD)	0.099	UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.099	U (LOD)	0.1	U (LOD)	0.098	U (LOD)	ND	ND	0.096	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.2	UJ (LOD)	0.2	UJ (LOD)	0.2	UJ (LOD)	ND	ND	0.19	UJ (LOD)	0.19	UJ (LOD)	0.2	UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.69	U (LOD)	0.7	U (LOD)	0.69	U (LOD)	ND	ND	0.67	U (LOD)	0.67	U (LOD)	0.69	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	390	5,700	mg/kg	0.069	U (LOD)	0.07	U (LOD)	0.069	U (LOD)	ND	ND	0.067	U (LOD)	0.067	U (LOD)	0.069	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.99	U (LOD)	1	U (LOD)	0.98	U (LOD)	ND	ND	0.96	U (LOD)	0.96	U (LOD)	0.99	U (LOD)	ND	ND
Tetryl	479-45-8	0.16	230	mg/kg	0.099	UJ (LOD)	0.1	UJ (LOD)	0.098	UJ (LOD)	ND	ND	0.096	UJ (LOD)	0.096	UJ (LOD)	0.099	UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 5b DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-17 (AOC #2 - Former Building 121 [Incinerator], AOC #16 - Area D - Dumping Area, and Previous Sampling Locations IX-121-B1 and II-EA-SD-02-02										
			Sample ID	IINY-DU-17A-SSIS-20200331	IINY-DU-17B-SSIS-20200331	IINY-DU-17C-SSIS-20200331	%RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-17A-SBIS1-20200331	IINY-DU-17B-SBIS1-20200331	IINY-DU-17C-SBIS1-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
			Sample Date	3/31/2020 8:15	3/31/2020 8:15	3/31/2020 8:15			3/31/2020 8:45	3/31/2020 8:45	3/31/2020 8:45		
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit										
Soil pH													
pH	N/A	N/A	N/A	7.2	7.2	7.1	0.806	7.31	7.0	7.4	7.7	4.77	8.25
Mercury													
Mercury	7439-97-6	0.013	mg/kg	0.98	0.61	0.77	24	1.25	0.9	0.8	0.77	8	0.995
Target Analyte List Metals													
Aluminum	7429-90-5	NS	mg/kg	5,400	5,300	7,000	16	8,300	6,100	5,900	6,100	2	6,320
Antimony	7440-36-0	0.27	mg/kg	9.9	7.3	6.5	22.5	12.4	4.8	4.5	4.4	4.56	5.09
Arsenic	7440-38-2	6.8	mg/kg	8.2	2.7	8.8	51.2	15	22	25	23	6.55	27.2
Barium	7440-39-3	110	mg/kg	150	120	130	11.5	172	460	330	280	26.1	590
Beryllium	7440-41-7	2.5	mg/kg	0.38	0.15	0.53	54.2	0.835	1.2	1.3	1.5	11.5	1.72
Cadmium	7440-43-9	0.36	mg/kg	5	20	16	56.8	33.2	3.9	3.4	3.2	10.3	4.41
Calcium	7440-70-2	NS	mg/kg	74,000	43,000	58,000	26.6	97300	14,000	19,000	22,000	22	28,500
Chromium, Total	7440-47-3	23	mg/kg	19	6.9	25	54.3	40.2	13	13	12	4.56	14.1
Cobalt	7440-48-4	13	mg/kg	7.2	4.9	5.7	19.7	8.87	6.3	6.8	6.6	3.83	7.2
Copper	7440-50-8	28	mg/kg	210	83	280	52.3	442	180	130	130	19.7	219
Iron	7439-89-6	NS	mg/kg	16,000	14,000	19,000	15.4	22,700	26,000	26,000	25,000	2.25	27,100
Lead	7439-92-1	11	mg/kg	2,900	1,500	1,200	48.6	4,150	2,700	1,200	820	63.2	4,070
Magnesium	7439-95-4	NS	mg/kg	45,000	26,000	34,000	27.3	59,000	6,400	8,500	9,800	20.8	12,600
Manganese	7439-96-5	220	mg/kg	400	380	440	7.51	484	170	170	170	0	170
Nickel	7440-02-0	38	mg/kg	16	5.9	20	52	32.3	19	21	19	5.87	22.6
Potassium	2023695	NS	mg/kg	1100	1100	1400	14.4	1640	610	550	560	5.61	654
Selenium	7782-49-2	0.52	mg/kg	0.52	0.2 J	0.68	52.4	1.08	1.1	1.4	1.5	15.6	1.86
Silver	7440-22-4	4.2	mg/kg	0.59	0.56	0.72	13.6	0.837	0.3	0.25	0.24	12.2	0.344
Sodium	7440-23-5	NS	mg/kg	400 J	370 J	490	14.9	577	510	480	510	3.46	544
Thallium	7440-28-0	0.05	mg/kg	0.12	0.1	0.15	20.4	0.187	0.6	0.72	0.8	14.2	0.96
Vanadium	7440-62-2	7.8	mg/kg	17	18	22	13.9	25.7	24	22	21	6.84	26.2
Zinc	7440-66-6	46	mg/kg	8,200	7,200	5,700	17.9	10,200	1,900	2,900	1,600	31.9	3,850
Polycyclic Aromatic Hydrocarbons													
1-Methylnaphthalene	90-12-0	29	mg/kg	0.03	0.026	0.037	18	0.045	0.087 J	0.098 J	0.11 J	11.7	0.127
2-Methylnaphthalene	91-57-6	29	mg/kg	0.044	0.039	0.056	18.900	0.0683	0.12 J	0.14 J	0.16 J	14.300	0.19
Acenaphthene	83-32-9	29	mg/kg	0.035	0.028	0.044	22.500	0.0559	0.068 J	0.058 J	0.087 J	20.700	0.108
Acenaphthylene	208-96-8	29	mg/kg	0.13	0.11	0.18	25.800	0.231	0.031 J	0.035 J	0.042 J	15.500	0.05
Anthracene	120-12-7	29	mg/kg	0.17	0.15	0.24	25.300	0.306	0.21	0.081 J	0.16 J	43.300	0.314
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.58	0.48 J	0.89	32.900	1.19	0.7	0.2 J	0.47 J	54.800	1.09
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.5	0.41 J	0.75	31.800	0.997	0.45	0.11 J	0.31 J	58.900	0.72
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	0.88	0.71 J	1.3	31.500	1.73	0.97	0.32 J	0.67 J	49.800	1.47
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.32	0.29	0.45	24.100	0.567	0.34	0.13 J	0.27 J	43.300	0.516
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.28	0.24	0.38	24.000	0.481	0.23 J	0.051 J	0.17 J	60.600	0.38
Chrysene	218-01-9	1.1	mg/kg	0.66	0.57 J	1	30.500	1.31	1	0.48 J	0.8 J	34.500	1.42
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.12	0.093	0.15	23.600	0.193	0.15 J	0.068 J	0.13 J	36.900	0.224
Fluoranthene	206-44-0	1.1	mg/kg	1.1	0.94 J	1.6	28.400	2.08	1.5	0.44 J	1 J	54.100	2.31
Fluorene	86-73-7	29	mg/kg	0.043	0.035	0.06	27.800	0.0781	0.076 J	0.064 J	0.11 J	28.600	0.143
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.38	0.3 J	0.52	27.800	0.68	0.29	0.085 J	0.25 J	52.200	0.482
Naphthalene	91-20-3	29	mg/kg	0.073	0.069	0.094	17.100	0.112	0.2 J	0.24 J	0.28 J	16.700	0.341
Phenanthrene	85-01-8	29	mg/kg	0.49	0.4 J	0.74	32.400	0.987	1.1	0.48 J	0.88 J	38.300	1.61
Pyrene	129-00-0	1.1	mg/kg	1.1	0.92 J	1.6	29.200	2.09	1.2	0.4 J	0.87 J	48.800	1.84
Total LMW PAHs	N/A	29	mg/kg	1.015	0.857	1.451	27.800	1.88	1.892	1.196	1.829	23.500	2.61
Total HMW PAHs	N/A	1.1	mg/kg	5.92	4.953	8.64	29.400	11.3	6.83	2.284	4.94	48.700	10.4
Polychlorinated Biphenyls													
PCB, Total	1336-36-3	0.041	mg/kg	0.14	0.13	0.14	4.22	0.151	0.016 UJ (LOD)	0.015 UJ (LOD)	0.015 UJ (LOD)	ND	ND
PCB-1016 (Aroclor 1016)	12674-11-2	0.041	mg/kg	0.03 U (LOD)	0.027 U (LOD)	0.027 U (LOD)	ND	ND	0.031 UJ (LOD)	0.029 UJ (LOD)	0.028 UJ (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.041	mg/kg	0.078 U (LOD)	0.071 U (LOD)	0.071 U (LOD)	ND	ND	0.082 UJ (LOD)	0.075 UJ (LOD)	0.073 UJ (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.041	mg/kg	0.03 U (LOD)	0.027 U (LOD)	0.027 U (LOD)	ND	ND	0.031 UJ (LOD)	0.029 UJ (LOD)	0.028 UJ (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.041	mg/kg	0.047 U (LOD)	0.043 U (LOD)	0.043 U (LOD)	ND	ND	0.049 UJ (LOD)	0.045 UJ (LOD)	0.044 UJ (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.041	mg/kg	0.095	0.084	0.087	6.41	0.103	0.015 UJ (LOD)	0.014 UJ (LOD)	0.014 UJ (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.041	mg/kg	0.032 U (LOD)	0.03 U (LOD)	0.03 U (LOD)	ND	ND	0.034 UJ (LOD)	0.031 UJ (LOD)	0.03 UJ (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.041	mg/kg	0.044 J	0.041 J	0.048 J	7.92	0.0532	0.0068 UJ (LOD)	0.0063 UJ (LOD)	0.0031 J	37.2	0.0105
PCB-1262 (Aroclor 1262)	37324-23-5	0.041	mg/kg	0.016 U (LOD)	0.014 U (LOD)	0.014 U (LOD)	ND	ND	0.016 UJ (LOD)	0.015 UJ (LOD)	0.015 UJ (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	0.041	mg/kg	0.0065 UJ (LOD)	0.0059 UJ (LOD)	0.0059 UJ (LOD)	ND	ND	0.0068 UJ (LOD)	0.0063 UJ (LOD)	0.0061 UJ (LOD)	ND	ND
Explosives Residues													
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.038 U (LOD)	0.037 UJ (LOD)	0.037 U (LOD)	ND	ND	0.038 U (LOD)	0.038 U (LOD)	0.038 U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.096 UJ (LOQ)	0.093 UJ (LOQ)	0.092 UJ (LOQ)	ND	ND	0.095 UJ (LOQ)	0.095 UJ (LOQ)	0.096 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.038 UJ (LOD)	0.037 UJ (LOD)	0.037 UJ (LOD)	ND	ND	0.038 UJ (LOD)	0.038 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.096 U (LOQ)	0.093 UJ (LOQ)	0.092 U (LOQ)	ND	ND	0.095 U (LOQ)	0.095 U (LOQ)	0.096 UJ (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.096 U (LOD)	0.093 UJ (LOD)	0.092 U (LOD)	ND	ND	0.095 U (LOD)	0.095 U (LOD)	0.096 U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.019 U (LOD)	0.019 UJ (LOD)	0.018 U (LOD)	ND	ND	0.019 U (LOD)	0.019 U (LOD)	0.019 U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.18 UJ (LOQ)	ND	ND	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.096 U (LOQ)	0.093 UJ (LOQ)	0.092 U (LOQ)	ND	ND	0.095 U (LOQ)	0.095 U (LOQ)	0.096 U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.096 UJ (LOD)	0.093 UJ (LOD)	0.092 UJ (LOD)	ND	ND	0.095 UJ (LOD)	0.095 UJ (LOD)	0.096 UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.096 U (LOD)	0.093 U (LOD)	0.092 U (LOD)	ND	ND	0.095 U (LOD)	0.095 U (LOD)	0.096 U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.19 UJ (LOD)	0.19 UJ (LOD)	0.18 UJ (LOD)	ND	ND	0.19 UJ (LOD)	0.19 UJ (LOD)	0.19 UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.67 U (LOD)	0.65 UJ (LOD)	0.64 U (LOD)	ND	ND	0.67 U (LOD)	0.67 U (LOD)	0.67 U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	16	mg/kg	0.067 U (LOD)	0.065 UJ (LOD)	0.064 U (LOD)	ND	ND	0.067 U (LOD)	0.067 U (LOD)	0.067 U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.96 U (LOD)	0.93 UJ (LOD)	0.92 U (LOD)	ND	ND	0.95 U (LOD)	0.95 U (LOD)	0.96 U (LOD)	ND	ND
Tetryl	479-45-8	0.018	mg/kg	0.19 U (LOQ)	0.093 UJ (LOD)	0.092 UJ (LOD)	ND	ND	0.095 UJ (LOD)	0.095 UJ (LOD)	0.096 UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 5b DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-18 (AOC #16 - Area D - Dumping Area and Previous Sampling Locations IX DMP-B22 and IXDMP-B1)										
			Sample ID	IINY-DU-18A-SSIS-20200331	IINY-DU-18B-SSIS-20200331	IINY-DU-18C-SSIS-20200331	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-18A-SBIS1-20200331	IINY-DU-18B-SBIS1-20200331	IINY-DU-18B-SBIS1-20200331	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
			Sample Date	3/31/2020 13:45	3/31/2020 13:45	3/31/2020 13:45			3/31/2020 14:20	3/31/2020 14:20	3/31/2020 14:20		
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit										
Soil pH													
pH	N/A	N/A	N/A	7.5	8.0	7.8	3.24	8.4	8.1	8.1	8	0.716	0
Mercury													
Mercury	7439-97-6	0.013	mg/kg	4.3	2.3	1.5	53	6.33	0.71	2.1	0.71	68	3.19
Target Analyte List Metals													
Aluminum	7429-90-5	NS	mg/kg	11,000	11,000	6,500	27	16,000	4,300	4,800	5,900	16	7,060
Antimony	7440-36-0	0.27	mg/kg	6.5	6.2	6.9	5.38	7.42	11	13	5.5	39.5	19.6
Arsenic	7440-38-2	6.8	mg/kg	7.8	15	15	33	23.1	17	19	16	8.81	21.2
Barium	7440-39-3	110	mg/kg	360	230	240	26.1	459	330	310	270	10.1	380
Beryllium	7440-41-7	2.5	mg/kg	0.56	0.55	0.66	10.3	0.743	0.64	0.69	0.63	4.92	0.734
Cadmium	7440-43-9	0.36	mg/kg	5.2	4.2	4.5	11.1	5.92	7.6	24	4.7	86	38.3
Calcium	7440-70-2	NS	mg/kg	12,000	12,000	13,000	4.68	13,800	44,000	32,000	20,000	37.5	62,200
Chromium, Total	7440-47-3	23	mg/kg	14	33	240	131	411	33	32	29	6.64	35.6
Cobalt	7440-48-4	13	mg/kg	7.6	7	7.2	4.2	8.04	7.5	5	6.5	19.9	9.5
Copper	7440-50-8	28	mg/kg	40	1,700	1,000	91.2	3,010	5,500	5,600	5,100	4.9	6,070
Iron	7439-89-6	NS	mg/kg	35,000	35,000	27,000	14.3	44,000	43000	36000	33000	13.7	50,200
Lead	7439-92-1	11	mg/kg	1,500	1,400	1,400	4.03	1,580	2,900	2,000	2,400	18.5	3,570
Magnesium	7439-95-4	NS	mg/kg	7900	7800	7,900	0.734	8,010	24,000	17,000	11,000	37.5	33,700
Manganese	7439-96-5	220	mg/kg	540	550	450	10.7	652	480	450	510	6.25	555
Nickel	7440-02-0	38	mg/kg	16	29	30	31.2	44.7	79	64	52	20.8	99
Potassium	2023695	NS	mg/kg	2,300	2,300	920	43.3	3,850	480	590	1,100	45.7	1560
Selenium	7782-49-2	0.52	mg/kg	0.36 J	0.74	0.58	34.1	1.04	0.79	0.95	0.86	9.25	1.07
Silver	7440-22-4	4.2	mg/kg	0.92	0.63	0.85	18.9	1.18	1.5	2.1	0.83	43	3.08
Sodium	7440-23-5	NS	mg/kg	810	810	590	17.2	1060	420 J	430 J	430 J	1.35	441
Thallium	7440-28-0	0.05	mg/kg	0.13	0.21	0.2	24.2	0.29	0.35	0.31	0.27	12.9	0.411
Vanadium	7440-62-2	7.8	mg/kg	29	24	29 J	10.6	34.6	17	18	18	3.27	19.1
Zinc	7440-66-6	46	mg/kg	83	1,800	1,600	80.9	3,520	5,600	5,000	3,100	28.6	7,850
Polycyclic Aromatic Hydrocarbons													
1-Methylnaphthalene	90-12-0	29	mg/kg	0.04	0.04 J	0.041 J	1.43	0.0418	0.1 J	0.08 J	0.086 J	11.6	0.114
2-Methylnaphthalene	91-57-6	29	mg/kg	0.07	0.06 J	0.072 J	9.550	0.0835	0.12 J	0.095 J	0.1 J	12.600	0.138
Acenaphthene	83-32-9	29	mg/kg	0.046	0.055 J	0.052 J	8.990	0.0625	0.23 J	0.18 J	0.18 J	14.700	0.269
Acenaphthylene	208-96-8	29	mg/kg	0.059	0.074 J	0.12 J	37.700	0.164	0.041 J	0.063 J	0.066 J	24.100	0.091
Anthracene	120-12-7	29	mg/kg	0.13	0.17 J	0.21 J	23.500	0.271	0.12 J	0.16 J	0.23 J	32.800	0.31
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.49 J	0.63 J	1	37.300	1.37	0.28 J	0.44 J	0.79	51.800	1.16
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.44 J	0.58 J	0.92	38.200	1.27	0.15 J	0.28 J	0.64	71.200	0.995
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	0.86 J	1.1 J	1.6	31.800	2.14	0.44 J	0.77 J	1.3	51.900	1.93
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.3	0.41 J	0.64	38.600	0.887	0.14 J	0.24 J	0.51	64.500	0.778
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.21	0.31 J	0.45	37.300	0.627	0.075 J	0.14 J	0.28	63.500	0.429
Chrysene	218-01-9	1.1	mg/kg	0.86 J	1.1 J	1.5	28.000	1.97	0.65 J	1.1 J	1.7	45.800	2.48
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.11	0.16 J	0.19 J	26.400	0.255	0.064 J	0.1 J	0.2 J	58.100	0.299
Fluoranthene	206-44-0	1.1	mg/kg	0.97 J	1.2 J	1.7	28.900	2.23	0.72 J	0.93 J	1.4	34.200	1.89
Fluorene	86-73-7	29	mg/kg	0.061	0.071 J	0.068 J	7.700	0.0796	0.15 J	0.14 J	0.16 J	6.670	0.175
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.31	0.45 J	0.71	41.400	1	0.1 J	0.22 J	0.45	69.300	0.704
Naphthalene	91-20-3	29	mg/kg	0.24	0.2 J	0.24 J	10.200	0.285	0.71 J	0.46 J	0.51	23.600	0.893
Phenanthrene	85-01-8	29	mg/kg	0.62 J	0.78 J	0.9	18.300	1.12	0.66 J	0.86 J	1.4	39.300	1.94
Pyrene	129-00-0	1.1	mg/kg	0.87 J	1.1 J	1.7	35.000	2.3	0.62 J	0.89 J	1.3	36.600	1.8
Total LMW PAHs	N/A	29	mg/kg	1.266	1.45	1.703	14.900	2.03	2.131	2.038	2.732	16.400	3.25
Total HMW PAHs	N/A	1.1	mg/kg	5.42	7.04	10.41	33.400	14	3.239	5.11	8.57	48.000	12.4
Polychlorinated Biphenyls													
PCB, Total	1336-36-3	0.041	mg/kg	0.29	0.17	0.4 J	40.1	0.576	0.15	0.1	0.057 J	45.5	0.219
PCB-1016 (Aroclor 1016)	12674-11-2	0.041	mg/kg	0.029 U (LOD)	0.028 U (LOD)	0.029 UJ (LOD)	ND	ND	0.03 U (LOD)	0.028 U (LOD)	0.029 U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.041	mg/kg	0.075 U (LOD)	0.074 U (LOD)	0.076 UJ (LOD)	ND	ND	0.078 U (LOD)	0.072 U (LOD)	0.075 U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.041	mg/kg	0.029 U (LOD)	0.028 U (LOD)	0.029 UJ (LOD)	ND	ND	0.03 U (LOD)	0.028 U (LOD)	0.029 U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.041	mg/kg	0.045 U (LOD)	0.044 U (LOD)	0.046 UJ (LOD)	ND	ND	0.047 U (LOD)	0.043 U (LOD)	0.045 U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.041	mg/kg	0.13	0.099	0.14 J	17.4	0.177	0.015 U (LOD)	0.013 U (LOD)	0.014 U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.041	mg/kg	0.031 U (LOD)	0.031 U (LOD)	0.032 UJ (LOD)	ND	ND	0.032 U (LOD)	0.03 U (LOD)	0.031 U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.041	mg/kg	0.16	0.074	0.25 J	54.6	0.383	0.0065 U (LOD)	0.006 U (LOD)	0.0062 U (LOD)	ND	ND
PCB-1262 (Aroclor 1262)	37324-23-5	0.041	mg/kg	0.015 U (LOD)	0.015 U (LOD)	0.015 UJ (LOD)	ND	ND	0.15 J	0.1 J	0.057 J	45.5	0.219
PCB-1268 (Aroclor 1268)	11100-14-4	0.041	mg/kg	0.0063 UJ (LOD)	0.0061 UJ (LOD)	0.0063 UJ (LOD)	ND	ND	0.0065 U (LOD)	0.006 U (LOD)	0.0062 U (LOD)	ND	ND
Explosives Residues													
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.037 U (LOD)	0.039 U (LOD)	0.038 U (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.094 UJ (LOQ)	0.098 UJ (LOQ)	0.096 UJ (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.037 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND	0.039 UJ (LOD)	0.039 UJ (LOD)	0.038 UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.094 UJ (LOQ)	0.098 UJ (LOQ)	0.096 UJ (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.094 U (LOD)	0.098 U (LOD)	0.096 U (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.019 U (LOD)	0.02 U (LOD)	0.019 U (LOD)	ND	ND	0.019 UJ (LOD)	0.019 UJ (LOD)	0.019 UJ (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.19 UJ (LOQ)	0.2 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND	0.19 UJ (LOQ)	0.19 UJ (LOQ)	0.19 UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.094 U (LOQ)	0.098 U (LOQ)	0.096 U (LOQ)	ND	ND	0.096 UJ (LOQ)	0.096 UJ (LOQ)	0.094 UJ (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.094 UJ (LOD)	0.098 UJ (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.094 UJ (LOD)	0.098 U (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.19 UJ (LOD)	0.2 UJ (LOD)	0.19 UJ (LOD)	ND	ND	0.19 UJ (LOD)	0.19 UJ (LOD)	0.19 UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.65 U (LOD)	0.68 U (LOD)	0.67 U (LOD)	ND	ND	0.68 UJ (LOD)	0.67 UJ (LOD)	0.66 UJ (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	16	mg/kg	0.065 U (LOD)	0.068 U (LOD)	0.067 U (LOD)	ND	ND	0.068 UJ (LOD)	0.067 UJ (LOD)	0.066 UJ (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.94 U (LOD)	0.98 U (LOD)	0.96 U (LOD)	ND	ND	0.96 UJ (LOD)	0.96 UJ (LOD)	0.94 UJ (LOD)	ND	ND
Tetryl	479-45-8	0.018	mg/kg	0.094 UJ (LOD)	0.098 UJ (LOD)	0.096 UJ (LOD)	ND	ND	0.096 UJ (LOD)	0.096 UJ (LOD)	0.094 UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 5b DU-17 Through DU-19 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-19 (AOC #16 - Area D - Dumping Area and Previous Sampling Location II-EA-SS-02-16															
			Sample ID	IINY-DU-19A-SSIS-20200331		IINY-DU-19B-SSIS-20200331		IINY-DU-19C-SSIS-20200331		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-19A-SBIS1-20200331		IINY-DU-19B-SBIS1-20200331		IINY-DU-19B-SBIS1-20200331		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6		0-6		0-6				6-36		6-36		6-36			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Subsurface Soil		Subsurface Soil		Subsurface Soil			
			Sample Date	3/31/2020 10:45		3/31/2020 10:45		3/31/2020 10:45				3/31/2020 11:00		3/31/2020 11:00		3/31/2020 11:00			
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit																
Soil pH																			
pH	N/A	N/A	N/A	7.7		7.5		7.4		2.03	7.92	0.12		0.23		0.27		38	0.402
Mercury																			
Mercury	7439-97-6	0.013	mg/kg	0.46	J	0.11	J	0.43	J	58	0.822	0.12		0.23		0.27		38	0.402
Target Analyte List Metals																			
Aluminum	7429-90-5	NS	mg/kg	16,000		25,000		20,000		22	31,700	25,000		20,000		21,000		12	28,700
Antimony	7440-36-0	0.27	mg/kg	0.5	J	0.28	J	0.5	J	29.8	0.746	0.12	J	0.11	J	0.11	J	5.09	0.128
Arsenic	7440-38-2	6.8	mg/kg	9.6	J	6.8	J	12	J	27.5	16	2.8		2.9		2.6		5.52	3.15
Barium	7440-39-3	110	mg/kg	81	J	63	J	83	J	14.6	103	34	J	37		35		4.32	39.2
Beryllium	7440-41-7	2.5	mg/kg	0.42	J	0.35	J	0.45	J	12.6	0.536	0.19		0.19		0.17		6.3	0.212
Cadmium	7440-43-9	0.36	mg/kg	0.33		0.25		0.3		13.8	0.395	0.097	J	0.093	J	0.087	J	5.45	0.105
Calcium	7440-70-2	NS	mg/kg	10,000	J	15,000		12,000		20	18,700	20,000		17,000		17,000		10	22,400
Chromium, Total	7440-47-3	23	mg/kg	10	J	10	J	11	J	5.59	11.8	7.8		7.7		7.8		0.743	7.91
Cobalt	7440-48-4	13	mg/kg	14		19		15		16.5	22.7	18		16		17		5.88	19.5
Copper	7440-50-8	28	mg/kg	130		180		140		17.6	217	210		210		210		0	210
Iron	7439-89-6	NS	mg/kg	23,000	J	36,000		29,000		22	45,700	36,000		33,000		35,000		4	38,500
Lead	7439-92-1	11	mg/kg	61		38		67		27.7	93.9	14		12		11		12.4	16.2
Magnesium	7439-95-4	NS	mg/kg	5,700	J	8,300	J	6,700	J	19	10,200	7,600		7,200		7,000		4	8,040
Manganese	7439-96-5	220	mg/kg	410	J	410	J	470	J	8.06	517	270	J	280		280		2.09	291
Nickel	7440-02-0	38	mg/kg	16	J	17	J	17	J	3.46	18.1	14		14		14		0	14
Potassium	2023695	NS	mg/kg	1,200		1,500		1,600		15	1,960	1,200		1,100		1,100		5	1,280
Selenium	7782-49-2	0.52	mg/kg	0.42	J	0.35	J	0.44	J	11.7	0.522	0.23	J	0.23	J	0.23	J	0	0.23
Silver	7440-22-4	4.2	mg/kg	0.12		0.12		0.13		4.68	0.138	0.11		0.097	J	0.1		6.65	0.119
Sodium	7440-23-5	NS	mg/kg	1,700		3,100		2,100		31	4,110	3,400		2,400		2,400		21	4,190
Thallium	7440-28-0	0.05	mg/kg	0.12	J	0.12	J	0.14	J	9.12	0.156	0.07	J	0.076	J	0.071	J	4.44	0.0804
Vanadium	7440-62-2	7.8	mg/kg	46		52		44		8.8	57.8	59		55		52		6.35	64.2
Zinc	7440-66-6	46	mg/kg	120	J	90	J	110	J	14.3	145	60		61		62		1.64	63.5
Polycyclic Aromatic Hydrocarbons																			
1-Methylnaphthalene	90-12-0	29	mg/kg	0.023		0.012		0.017		31.8	0.0312	0.0011	J	0.0012	J	0.00064	J	30.5	0.00173
2-Methylnaphthalene	91-57-6	29	mg/kg	0.035	J	0.018	J	0.028	J	31.600	0.0485	0.0017	J	0.0019	J	0.00093	J	33.900	0.0028
Acenaphthene	83-32-9	29	mg/kg	0.06		0.026		0.027		51.400	0.0864	0.0024	J	0.0015	J	0.00087	J	48.400	0.00353
Acenaphthylene	208-96-8	29	mg/kg	0.27	J	0.13	J	0.21	J	34.500	0.38	0.0066	J	0.0069	J	0.0034	J	34.400	0.0105
Anthracene	120-12-7	29	mg/kg	0.43		0.19		0.24		44.200	0.605	0.0067	J	0.0064	J	0.0032	J	35.700	0.0103
Benzo(a)anthracene	56-55-3	1.1	mg/kg	2.5		1.1		1.1		51.600	3.6	0.024		0.029		0.013		37.200	0.0426
Benzo(a)pyrene	50-32-8	1.1	mg/kg	1.9		0.9		0.98		44.100	2.66	0.029		0.031		0.015		34.900	0.0469
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	3.4		1.6		1.8		43.500	4.75	0.045		0.047		0.023		34.700	0.0718
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	1.1		0.55		0.64		38.600	1.51	0.025		0.028		0.015		30.000	0.0398
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	1.2		0.59		0.59		44.400	1.68	0.013		0.015		0.0072	J	34.500	0.0219
Chrysene	218-01-9	1.1	mg/kg	2.3		0.98		1.1		50.000	3.3	0.027		0.033		0.014		39.400	0.0491
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.41		0.26		0.27		26.800	0.524	0.0061	J	0.0067	J	0.0039	J	26.500	0.00928
Fluoranthene	206-44-0	1.1	mg/kg	4		1.6		1.7		55.800	5.85	0.046		0.053		0.023		38.600	0.0802
Fluorene	86-73-7	29	mg/kg	0.077	J	0.028	J	0.039	J	53.600	0.113	0.0037	J	0.0028	J	0.0011	J	52.100	0.00586
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	1.5		0.77		0.82		39.600	2.06	0.027		0.029		0.014		34.900	0.0438
Naphthalene	91-20-3	29	mg/kg	0.083	J	0.046	J	0.068	J	28.300	0.113	0.0019	J	0.0022	J	0.0019	U (LOD)	8.660	0.00244
Phenanthrene	85-01-8	29	mg/kg	1.5		0.52		0.58		63.400	2.25	0.022		0.023		0.0091	J	43.000	0.0375
Pyrene	129-00-0	1.1	mg/kg	3.2		1.3		1.4		54.400	4.66	0.044		0.052		0.023		37.800	0.0774
Total LMW PAHs	N/A	29	mg/kg	2.478		0.97		1.209		52.200	3.59	0.0461		0.0459		0.02114		38.100	0.0738
Total HMW PAHs	N/A	1.1	mg/kg	21.51		9.65		10.4		47.900	30.6	0.2861		0.3237		0.1511		35.800	0.482
Polychlorinated Biphenyls																			
PCB, Total	1336-36-3	0.041	mg/kg	0.011	J	0.0071	J	0.015	UJ (LOD)	35.8	0.021	0.05	J	0.049	J	0.054	J	5.19	0.0577
PCB-1016 (Aroclor 1016)	12674-11-2	0.041	mg/kg	0.029	U (LOD)	0.028	U (LOD)	0.029	UJ (LOD)	ND	ND	0.03	U (LOD)	0.029	U (LOD)	0.03	U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.041	mg/kg	0.076	U (LOD)	0.072	U (LOD)	0.075	UJ (LOD)	ND	ND	0.077	U (LOD)	0.076	U (LOD)	0.077	U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.041	mg/kg	0.029	U (LOD)	0.028	U (LOD)	0.029	UJ (LOD)	ND	ND	0.03	U (LOD)	0.029	U (LOD)	0.03	U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.041	mg/kg	0.046	U (LOD)	0.043	U (LOD)	0.045	UJ (LOD)	ND	ND	0.046	U (LOD)	0.046	U (LOD)	0.046	U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.041	mg/kg	0.014	U (LOD)	0.013	U (LOD)	0.014	UJ (LOD)	ND	ND	0.015	U (LOD)	0.014	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.041	mg/kg	0.032	U (LOD)	0.03	U (LOD)	0.031	UJ (LOD)	ND	ND	0.032	U (LOD)	0.032	U (LOD)	0.032	U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.041	mg/kg	0.011	J	0.0071	J	0.0022	J	65.2	0.0179	0.05	J	0.049	J	0.054	J	5.19	0.0577
PCB-1262 (Aroclor 1262)	37324-23-5	0.041	mg/kg	0.015	U (LOD)	0.014	U (LOD)	0.015	UJ (LOD)	ND	ND	0.015	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	0.041	mg/kg	0.0063	U (LOD)	0.006	U (LOD)	0.0063	UJ (LOD)	ND	ND	0.0065	U (LOD)	0.0064	U (LOD)	0.0065	U (LOD)	ND	ND
Explosives Residues																			
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.038	U (LOD)	0.04	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.099	UJ (LOQ)	0.1	UJ (LOQ)	0.098	UJ (LOQ)	ND	ND	0.096	UJ (LOQ)	0.096	UJ (LOQ)	0.099	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.039	UJ (LOD)	0.04	UJ (LOD)	0.039	UJ (LOD)	ND	ND	0.038	UJ (LOD)	0.038	UJ (LOD)	0.04	UJ (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.099	U (LOQ)	0.1	U (LOQ)	0.098	U (LOQ)	ND	ND	0.096	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.099	U (LOD)	0.1	U (LOD)	0.098	U (LOD)	ND	ND	0.096	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.02	UJ (LOD)	0.02	UJ (LOD)	0.02	UJ (LOD)	ND	ND	0.019	U (LOD)	0.019	U (LOD)	0.02	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.2	UJ (LOQ)	0.2	UJ (LOQ)	0.2	UJ (LOQ)	ND	ND	0.19	UJ (LOQ)	0.19	UJ (LOQ)	0.2	UJ (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.099	U (LOQ)	0.1	U (LOQ)	0.098	U (LOQ)	ND	ND	0.096	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.099	UJ (LOD)	0.1	UJ (LOD)	0.098	UJ (LOD)	ND	ND	0.096	UJ (LOD)	0.096	UJ (LOD)	0.099	UJ (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.099	U (LOD)	0.1	U (LOD)	0.098	U (LOD)	ND	ND	0.096	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.2	UJ (LOD)	0.2	UJ (LOD)	0.2	UJ (LOD)	ND	ND	0.19	UJ (LOD)	0.19	UJ (LOD)	0.2	UJ (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.69	U (LOD)	0.7	U (LOD)	0.69	U (LOD)	ND	ND	0.67	U (LOD)	0.67	U (LOD)	0.69	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	16	mg/kg	0.069	U (LOD)	0.07	U (LOD)	0.069	U (LOD)	ND	ND	0.067	U (LOD)	0.067	U (LOD)	0.069	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.99	U (LOD)	1	U (LOD)	0.98	U (LOD)	ND	ND	0.96	U (LOD)	0.96	U (LOD)	0.99	U (LOD)	ND	ND
Tetryl	479-45-8	0.018	mg/kg	0.099	UJ (LOD)	0.1	UJ (LOD)	0.098	UJ (LOD)	ND	ND	0.096	UJ (LOD)	0.096	UJ (LOD)	0.099	UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 6a DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-20 (AOC #13: Area A – One Former Fuel Oil AST [2,500 gallons] Located Near Former Building 223)										
				Sample ID	IINY-DU-20A-SSIS-20200316	IINY-DU-20B-SSIS-20200316	IINY-DU-20C-SSIS-20200316	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-20A-SBIS1-20200317	IINY-DU-20B-SBIS1-20200317	IINY-DU-20C-SBIS1-20200317	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
				Sample Date	3/16/2020 15:30	3/16/2020 15:30	3/16/2020 15:30			3/17/2020 9:30	3/17/2020 9:30	3/17/2020 9:30		
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**										
Soil pH														
pH	N/A	N/A	N/A	N/A	6.1	6.5	6.6	4.13	7.07	6.8	6.9	6.8	0.845	6.98
Polycyclic Aromatic Hydrocarbons														
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.0076 J	0.011	0.0076 J	22.5	0.0137	0.0049 J	0.005 J	0.0049 J	1.17	0.00508
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.011	0.018	0.01	33.500	0.024	0.0078 J	0.0069 J	0.008 J	7.740	0.00904
Acenaphthene	83-32-9	360	4,500	mg/kg	0.0063 J	0.016	0.012	42.600	0.0237	0.0067 J	0.0079 J	0.0035 J	37.700	0.0118
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.029	0.041	0.032	18.400	0.0497	0.02	0.017	0.027	24.100	0.0342
Anthracene	120-12-7	1,800	23,000	mg/kg	0.035	0.046	0.043	13.800	0.0556	0.025	0.028	0.025	6.660	0.0304
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.14	0.15	0.15	3.940	0.161	0.074	0.074	0.071	2.370	0.0774
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.16	0.17	0.15	6.250	0.185	0.074	0.071	0.079	5.410	0.0848
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	0.25	0.28	0.23	9.930	0.317	0.11	0.11	0.12	5.090	0.128
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.13	0.13	0.11	9.360	0.152	0.045	0.042	0.05	8.850	0.0558
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.081	0.093	0.072	12.800	0.109	0.033	0.032	0.036	6.180	0.0389
Chrysene	218-01-9	110	2,100	mg/kg	0.17	0.19	0.16	8.810	0.212	0.079	0.076	0.083	4.430	0.0882
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.03	0.028	0.028	4.030	0.0316	0.013	0.013	0.015	8.450	0.0166
Fluoranthene	206-44-0	240	3,000	mg/kg	0.27	0.32	0.28	9.120	0.357	0.14 J	0.14 J	0.13 J	4.220	0.151
Fluorene	86-73-7	240	3,000	mg/kg	0.0098 J	0.02	0.016	33.700	0.0282	0.0087 J	0.011	0.0072 J	21.300	0.0138
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.13	0.15	0.12	11.500	0.172	0.049	0.045	0.053	8.160	0.0591
Naphthalene	91-20-3	2	8.6	mg/kg	0.012	0.017	0.011	24.100	0.0214	0.0072 J	0.007 J	0.0083 J	9.330	0.00926
Phenanthrene	85-01-8	NS	NS	mg/kg	0.1	0.14	0.13	16.900	0.176	0.072	0.081	0.056	18.200	0.102
Pyrene	129-00-0	180	2,300	mg/kg	0.25	0.31	0.27	11.000	0.354	0.14 J	0.14 J	0.14 J	0.000	0.14
Total LMW PAHs	N/A	NS	NS	mg/kg	0.2107	0.309	0.2616	18.900	0.384	0.1523	0.1638	0.1399	7.860	0.182
Total HMW PAHs	N/A	NS	NS	mg/kg	1.611	1.821	1.57	8.080	2.01	0.757	0.743	0.777	2.250	0.802
Benzene, Toluene, Ethylbenzene, and Xylenes														
Benzene	71-43-2	1.2	5.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0018 U (LOD)	0.0015 U (LOD)	0.0017 U (LOD)	ND	ND
Ethylbenzene	100-41-4	5.8	25	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
m,p-Xylene	108-38-3	55	240	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0018 U (LOD)	0.0015 U (LOD)	0.0017 U (LOD)	ND	ND
o-Xylene	95-47-6	65	280	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
Toluene	108-88-3	490	4,700	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
Xylenes, Total	1330-20-7	58	250	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 6a DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-21 (AOC #14: Area B – One Former Fuel Oil AST [Size Unknown] Located Near Dock 131)										
				Sample ID	IINY-DU-21A-SSIS-20200317	IINY-DU-21B-SSIS-20200317	IINY-DU-21C-SSIS-20200317	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-21A-SBIS1-20200317	IINY-DU-21B-SBIS1-20200317	IINY-DU-21C-SBIS1-20200317	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
				Sample Date	3/17/2020 12:40	3/17/2020 12:40	3/17/2020 12:40			3/17/2020 11:45	3/17/2020 11:45	3/17/2020 11:45		
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit										
Soil pH														
pH	N/A	N/A	N/A	N/A	6.3	6.8	6.8	4.35	7.36	7.0	7.1	7.3	2.14	7.52
Polycyclic Aromatic Hydrocarbons														
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.0083 J	0.0044 J	0.0041 J	41.8	0.0115	0.0063 J	0.0053 J	0.0052 J	10.9	0.00713
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.01	0.0067 J	0.0063 J	26.500	0.0128	0.0083 J	0.0081 J	0.0074 J	5.960	0.00912
Acenaphthene	83-32-9	360	4,500	mg/kg	0.025	0.0051 J	0.0049 J	99.000	0.0407	0.0067 J	0.004 J	0.0038 J	33.500	0.00891
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.028	0.019	0.021	20.800	0.0346	0.012	0.0098	0.012	11.300	0.0145
Anthracene	120-12-7	1,800	23,000	mg/kg	0.08	0.019	0.023	83.900	0.127	0.021	0.013	0.013	29.500	0.0273
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.26	0.088	0.098	64.900	0.392	0.073	0.061	0.066	9.040	0.0818
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.22	0.089	0.1	53.300	0.319	0.073	0.061	0.069	9.030	0.083
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	0.35	0.15	0.18	47.600	0.498	0.13	0.11	0.13	9.360	0.152
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.12	0.058	0.063	42.900	0.167	0.045	0.037	0.04	9.940	0.0508
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.12	0.05	0.057	51.000	0.173	0.043	0.036	0.036	10.500	0.0485
Chrysene	218-01-9	110	2,100	mg/kg	0.25	0.097	0.11	55.700	0.366	0.087	0.071	0.078	10.200	0.0989
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.038	0.016	0.019	49.000	0.0544	0.013	0.011	0.012	8.330	0.0145
Fluoranthene	206-44-0	240	3,000	mg/kg	0.47	0.16	0.18	64.300	0.707	0.15	0.11	0.12	16.400	0.179
Fluorene	86-73-7	240	3,000	mg/kg	0.03	0.0068 J	0.0077 J	88.600	0.0479	0.0074 J	0.0051 J	0.0046 J	26.200	0.00946
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.15	0.07	0.074	46.000	0.211	0.053	0.042	0.047	11.600	0.0612
Naphthalene	91-20-3	2	8.6	mg/kg	0.012	0.008 J	0.007 J	29.400	0.0157	0.0086 J	0.0076 J	0.0073 J	8.690	0.00955
Phenanthrene	85-01-8	NS	NS	mg/kg	0.32	0.058	0.066	101.000	0.523	0.077	0.047	0.046	31.100	0.101
Pyrene	129-00-0	180	2,300	mg/kg	0.5	0.17	0.18	66.200	0.756	0.15	0.11	0.12	16.400	0.179
Total LMW PAHs	N/A	NS	NS	mg/kg	0.5133	0.127	0.14	84.300	0.812	0.1473	0.0999	0.0993	23.800	0.185
Total HMW PAHs	N/A	NS	NS	mg/kg	2.478	0.948	1.061	57.000	3.64	0.817	0.649	0.718	11.600	0.941
Benzene, Toluene, Ethylbenzene, and Xylenes														
Benzene	71-43-2	1.2	5.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0026 U (LOD)	0.0018 U (LOD)	0.0026 U (LOD)	ND	ND
Ethylbenzene	100-41-4	5.8	25	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
m,p-Xylene	108-38-3	55	240	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0026 U (LOD)	0.0018 U (LOD)	0.0026 U (LOD)	ND	ND
o-Xylene	95-47-6	65	280	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
Toluene	108-88-3	490	4,700	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
Xylenes, Total	1330-20-7	58	250	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
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PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 6a DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-22 (AOC #15: Area C – Two Former Fuel Oil ASTs [Size Unknown] Located Near Former Building 417				
				Sample ID	IINY-DU-22A-SSIS-20200316	IINY-DU-22B-SSIS-20200316	IINY-DU-22C-SSIS-20200316	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/16/2020 16:45	3/16/2020 16:45	3/16/2020 16:45		
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**					
Soil pH									
pH	N/A	N/A	N/A	N/A	6.7	6.8	6.8		
Polycyclic Aromatic Hydrocarbons									
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.041	0.04	0.041	1.420	0.042
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.064	0.057	0.063	6.170	0.071
Acenaphthene	83-32-9	360	4,500	mg/kg	0.1	0.11	0.077	17.700	0.138
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.26 J	0.18	0.15	28.900	0.340
Anthracene	120-12-7	1,800	23,000	mg/kg	0.44	0.29	0.27	27.900	0.567
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	2	1.3	1.2	29.100	2.600
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	1.9	1.3	1.1	29.000	2.480
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	3.1	1.9	1.8	31.900	4.090
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	1	0.78	0.64	22.500	1.260
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	1.1	0.62	0.56	38.900	1.500
Chrysene	218-01-9	110	2,100	mg/kg	2.2	1.4	1.2	33.100	2.930
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.38	0.23	0.2	35.700	0.513
Fluoranthene	206-44-0	240	3,000	mg/kg	3.5	2.4	1.9	31.500	4.660
Fluorene	86-73-7	240	3,000	mg/kg	0.13	0.12	0.069	30.800	0.189
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	1.2	0.92	0.73	24.900	1.550
Naphthalene	91-20-3	2	8.6	mg/kg	0.066	0.053	0.063	11.200	0.078
Phenanthrene	85-01-8	NS	NS	mg/kg	1.4	1.1	0.71	32.300	1.940
Pyrene	129-00-0	180	2,300	mg/kg	3.5	2.4	2	29.500	4.590
Total LMW PAHs	N/A	NS	NS	mg/kg	2.501	1.95	1.443	26.900	3.3
Total HMW PAHs	N/A	NS	NS	mg/kg	19.88	13.25	11.33	30.300	26.1
Benzene, Toluene, Ethylbenzene, and Xylenes									
Benzene	71-43-2	1.2	5.1	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Ethylbenzene	100-41-4	5.8	25	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
m,p-Xylene	108-38-3	55	240	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
o-Xylene	95-47-6	65	280	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Toluene	108-88-3	490	4,700	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Xylenes, Total	1330-20-7	58	250	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 6b DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-20 (AOC #13: Area A – One Former Fuel Oil AST [2,500 gallons] Located Near Former Building 223)										
			Sample ID	IINY-DU-20A-SSIS-20200316	IINY-DU-20B-SSIS-20200316	IINY-DU-20C-SSIS-20200316	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-20A-SBIS1-20200317	IINY-DU-20B-SBIS1-20200317	IINY-DU-20C-SBIS1-20200317	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
			Sample Date	3/16/2020 15:30	3/16/2020 15:30	3/16/2020 15:30			3/17/2020 9:30	3/17/2020 9:30	3/17/2020 9:30		
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**										
Soil pH													
pH	N/A	N/A	N/A	6.1	6.5	6.6	4.13	7.07	6.8	6.9	6.8	0.845	6.98
Polycyclic Aromatic Hydrocarbons													
1-Methylnaphthalene	90-12-0	29	mg/kg	0.0076 J	0.011	0.0076 J	22.5	0.0137	0.0049 J	0.005 J	0.0049 J	1.17	0.00508
2-Methylnaphthalene	91-57-6	29	mg/kg	0.011	0.018	0.01	33.500	0.024	0.0078 J	0.0069 J	0.008 J	7.740	0.00904
Acenaphthene	83-32-9	29	mg/kg	0.0063 J	0.016	0.012	42.600	0.0237	0.0067 J	0.0079 J	0.0035 J	37.700	0.0118
Acenaphthylene	208-96-8	29	mg/kg	0.029	0.041	0.032	18.400	0.0497	0.02	0.017	0.027	24.100	0.0342
Anthracene	120-12-7	29	mg/kg	0.035	0.046	0.043	13.800	0.0556	0.025	0.028	0.025	6.660	0.0304
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.14	0.15	0.15	3.940	0.161	0.074	0.074	0.071	2.370	0.0774
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.16	0.17	0.15	6.250	0.185	0.074	0.071	0.079	5.410	0.0848
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	0.25	0.28	0.23	9.930	0.317	0.11	0.11	0.12	5.090	0.128
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.13	0.13	0.11	9.360	0.152	0.045	0.042	0.05	8.850	0.0558
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.081	0.093	0.072	12.800	0.109	0.033	0.032	0.036	6.180	0.0389
Chrysene	218-01-9	1.1	mg/kg	0.17	0.19	0.16	8.810	0.212	0.079	0.076	0.083	4.430	0.0882
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.03	0.028	0.028	4.030	0.0316	0.013	0.013	0.015	8.450	0.0166
Fluoranthene	206-44-0	1.1	mg/kg	0.27	0.32	0.28	9.120	0.357	0.14 J	0.14 J	0.13 J	4.220	0.151
Fluorene	86-73-7	29	mg/kg	0.0098 J	0.02	0.016	33.700	0.0282	0.0087 J	0.011	0.0072 J	21.300	0.0138
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.13	0.15	0.12	11.500	0.172	0.049	0.045	0.053	8.160	0.0591
Naphthalene	91-20-3	29	mg/kg	0.012	0.017	0.011	24.100	0.0214	0.0072 J	0.007 J	0.0083 J	9.330	0.00926
Phenanthrene	85-01-8	29	mg/kg	0.1	0.14	0.13	16.900	0.176	0.072	0.081	0.056	18.200	0.102
Pyrene	129-00-0	1.1	mg/kg	0.25	0.31	0.27	11.000	0.354	0.14 J	0.14 J	0.14 J	0.000	0.14
Total LMW PAHs	N/A	29	mg/kg	0.2107	0.309	0.2616	18.900	0.384	0.1523	0.1638	0.1399	7.860	0.182
Total HMW PAHs	N/A	1.1	mg/kg	1.611	1.821	1.57	8.080	2.01	0.757	0.743	0.777	2.250	0.802
Benzene, Toluene, Ethylbenzene, and Xylenes													
Benzene	71-43-2	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0018 U (LOD)	0.0015 U (LOD)	0.0017 U (LOD)	ND	ND
Ethylbenzene	100-41-4	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
m,p-Xylene	108-38-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0018 U (LOD)	0.0015 U (LOD)	0.0017 U (LOD)	ND	ND
o-Xylene	95-47-6	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
Toluene	108-88-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND
Xylenes, Total	1330-20-7	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0009 U (LOD)	0.00077 U (LOD)	0.00083 U (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 6b DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-21 (AOC #14: Area B – One Former Fuel Oil AST [Size Unknown] Located Near Dock 131)										
			Sample ID	IINY-DU-21A-SSIS-20200317	IINY-DU-21B-SSIS-20200317	IINY-DU-21C-SSIS-20200317	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-21A-SBIS1-20200317	IINY-DU-21B-SBIS1-20200317	IINY-DU-21C-SBIS1-20200317	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6	0-6	0-6			6-36	6-36	6-36		
			Matrix	Surface Soil	Surface Soil	Surface Soil			Subsurface Soil	Subsurface Soil	Subsurface Soil		
			Sample Date	3/17/2020 12:40	3/17/2020 12:40	3/17/2020 12:40			3/17/2020 11:45	3/17/2020 11:45	3/17/2020 11:45		
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**										
Soil pH													
pH	N/A	N/A	N/A	6.3	6.8	6.8	4.35	7.36	7.0	7.1	7.3	2.14	7.52
Polycyclic Aromatic Hydrocarbons													
1-Methylnaphthalene	90-12-0	29	mg/kg	0.0083 J	0.0044 J	0.0041 J	41.8	0.0115	0.0063 J	0.0053 J	0.0052 J	10.9	0.00713
2-Methylnaphthalene	91-57-6	29	mg/kg	0.01	0.0067 J	0.0063 J	26.500	0.0128	0.0083 J	0.0081 J	0.0074 J	5.960	0.00912
Acenaphthene	83-32-9	29	mg/kg	0.025	0.0051 J	0.0049 J	99.000	0.0407	0.0067 J	0.004 J	0.0038 J	33.500	0.00891
Acenaphthylene	208-96-8	29	mg/kg	0.028	0.019	0.021	20.800	0.0346	0.012	0.0098	0.012	11.300	0.0145
Anthracene	120-12-7	29	mg/kg	0.08	0.019	0.023	83.900	0.127	0.021	0.013	0.013	29.500	0.0273
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.26	0.088	0.098	64.900	0.392	0.073	0.061	0.066	9.040	0.0818
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.22	0.089	0.1	53.300	0.319	0.073	0.061	0.069	9.030	0.083
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	0.35	0.15	0.18	47.600	0.498	0.13	0.11	0.13	9.360	0.152
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.12	0.058	0.063	42.900	0.167	0.045	0.037	0.04	9.940	0.0508
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.12	0.05	0.057	51.000	0.173	0.043	0.036	0.036	10.500	0.0485
Chrysene	218-01-9	1.1	mg/kg	0.25	0.097	0.11	55.700	0.366	0.087	0.071	0.078	10.200	0.0989
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.038	0.016	0.019	49.000	0.0544	0.013	0.011	0.012	8.330	0.0145
Fluoranthene	206-44-0	1.1	mg/kg	0.47	0.16	0.18	64.300	0.707	0.15	0.11	0.12	16.400	0.179
Fluorene	86-73-7	29	mg/kg	0.03	0.0068 J	0.0077 J	88.600	0.0479	0.0074 J	0.0051 J	0.0046 J	26.200	0.00946
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.15	0.07	0.074	46.000	0.211	0.053	0.042	0.047	11.600	0.0612
Naphthalene	91-20-3	29	mg/kg	0.012	0.008 J	0.007 J	29.400	0.0157	0.0086 J	0.0076 J	0.0073 J	8.690	0.00955
Phenanthrene	85-01-8	29	mg/kg	0.32	0.058	0.066	101.000	0.523	0.077	0.047	0.046	31.100	0.101
Pyrene	129-00-0	1.1	mg/kg	0.5	0.17	0.18	66.200	0.756	0.15	0.11	0.12	16.400	0.179
Total LMW PAHs	N/A	29	mg/kg	0.5133	0.127	0.14	84.300	0.812	0.1473	0.0999	0.0993	23.800	0.185
Total HMW PAHs	N/A	1.1	mg/kg	**2.478**	0.948	1.061	57.000	**3.64**	0.817	0.649	0.718	11.600	0.941
Benzene, Toluene, Ethylbenzene, and Xylenes													
Benzene	71-43-2	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0026 U (LOD)	0.0018 U (LOD)	0.0026 U (LOD)	ND	ND
Ethylbenzene	100-41-4	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
m,p-Xylene	108-38-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0026 U (LOD)	0.0018 U (LOD)	0.0026 U (LOD)	ND	ND
o-Xylene	95-47-6	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
Toluene	108-88-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND
Xylenes, Total	1330-20-7	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A	0.0013 U (LOD)	0.00089 U (LOD)	0.0013 U (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 6b DU-20 Through DU-22 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-22 (AOC #15: Area C – Two Former Fuel Oil ASTs [Size Unknown] Located Near Former Building 417				
			Sample ID	IINY-DU-22A-SSIS-20200316	IINY-DU-22B-SSIS-20200316	IINY-DU-22C-SSIS-20200316	% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6	0-6	0-6		
			Matrix	Surface Soil	Surface Soil	Surface Soil		
			Sample Date	3/16/2020 16:45	3/16/2020 16:45	3/16/2020 16:45		
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**					
Soil pH								
pH	N/A	N/A	N/A	6.7	6.8	6.8		
Polycyclic Aromatic Hydrocarbons								
1-Methylnaphthalene	90-12-0	29	mg/kg	0.041	0.04	0.041	1.420	0.042
2-Methylnaphthalene	91-57-6	29	mg/kg	0.064	0.057	0.063	6.170	0.071
Acenaphthene	83-32-9	29	mg/kg	0.1	0.11	0.077	17.700	0.138
Acenaphthylene	208-96-8	29	mg/kg	0.26 J	0.18	0.15	28.900	0.340
Anthracene	120-12-7	29	mg/kg	0.44	0.29	0.27	27.900	0.567
Benzo(a)anthracene	56-55-3	1.1	mg/kg	2	1.3	1.2	29.100	2.600
Benzo(a)pyrene	50-32-8	1.1	mg/kg	1.9	1.3	1.1	29.000	2.480
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	3.1	1.9	1.8	31.900	4.090
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	1	0.78	0.64	22.500	1.260
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	1.1	0.62	0.56	38.900	1.500
Chrysene	218-01-9	1.1	mg/kg	2.2	1.4	1.2	33.100	2.930
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.38	0.23	0.2	35.700	0.513
Fluoranthene	206-44-0	1.1	mg/kg	3.5	2.4	1.9	31.500	4.660
Fluorene	86-73-7	29	mg/kg	0.13	0.12	0.069	30.800	0.189
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	1.2	0.92	0.73	24.900	1.550
Naphthalene	91-20-3	29	mg/kg	0.066	0.053	0.063	11.200	0.078
Phenanthrene	85-01-8	29	mg/kg	1.4	1.1	0.71	32.300	1.940
Pyrene	129-00-0	1.1	mg/kg	3.5	2.4	2	29.500	4.590
Total LMW PAHs	N/A	29	mg/kg	2.501	1.95	1.443	26.900	3.3
Total HMW PAHs	N/A	1.1	mg/kg	**19.88**	**13.25**	**11.33**	30.300	**26.1**
Benzene, Toluene, Ethylbenzene, and Xylenes								
Benzene	71-43-2	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Ethylbenzene	100-41-4	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
m,p-Xylene	108-38-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
o-Xylene	95-47-6	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Toluene	108-88-3	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A
Xylenes, Total	1330-20-7	NS	mg/kg	Not analyzed	Not analyzed	Not analyzed	N/A	N/A

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 7a DU-23 and DU-25 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	DU-23 (T1 Northwest of Building 406)							DU-25 (T3 Northeast of Building 220)							
				Sample ID	IINY-DU-23A-SSIS-20200320		IINY-DU-23B-SSIS-20200320		IINY-DU-23C-SSIS-20200320		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-25A-SSIS-20200320		IINY-DU-25B-SSIS-20200320		IINY-DU-25C-SSIS-20200320		% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6			
				Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
				Sample Date	3/20/2020 9:15		3/20/2020 9:15		3/20/2020 9:15				3/20/2020 10:30		3/20/2020 10:30		3/20/2020 10:30			
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**																
Soil pH																				
pH	N/A	N/A	N/A	N/A	6.0		5.4		5.5		5.71	6.44	6.2		6.4		6.4		1.82	6.62
Polychlorinated Biphenyls																				
PCB, Total	1336-36-3	0.23	0.94	mg/kg	0.034	J	0.025	J	0.019	J	29.000	0.045	0.011	J	0.016	J	0.012	J	20.400	0.020
PCB-1016 (Aroclor 1016)	12674-11-2	0.41	5.1	mg/kg	0.03	U (LOD)	0.03	U (LOD)	0.03	U (LOD)	ND	ND	0.03	U (LOD)	0.03	U (LOD)	0.029	U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.2	0.83	mg/kg	0.077	U (LOD)	0.077	U (LOD)	0.079	U (LOD)	ND	ND	0.078	U (LOD)	0.077	U (LOD)	0.076	U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.17	0.72	mg/kg	0.03	U (LOD)	0.03	U (LOD)	0.03	U (LOD)	ND	ND	0.03	U (LOD)	0.03	U (LOD)	0.029	U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.23	0.95	mg/kg	0.046	U (LOD)	0.046	U (LOD)	0.047	U (LOD)	ND	ND	0.047	U (LOD)	0.046	U (LOD)	0.046	U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.23	0.95	mg/kg	0.015	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND	0.015	U (LOD)	0.014	U (LOD)	0.014	U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.12	0.97	mg/kg	0.032	U (LOD)	0.032	U (LOD)	0.033	U (LOD)	ND	ND	0.032	U (LOD)	0.032	U (LOD)	0.032	U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.24	0.99	mg/kg	0.034	J	0.025	J	0.019	J	29.000	0.045	0.011	J	0.016	J	0.012	J	20.400	0.020
PCB-1262 (Aroclor 1262)	37324-23-5	NS	NS	mg/kg	0.015	U (LOD)	0.015	U (LOD)	0.016	U (LOD)	ND	ND	0.016	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	NS	NS	mg/kg	0.0065	U (LOD)	0.0065	U (LOD)	0.0066	U (LOD)	ND	ND	0.0065	U (LOD)	0.0064	U (LOD)	0.0063	U (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

DU-24 (T2 near AOC #16 – Area Dumping Area) was not sampled. The majority of DU-24 contained exposed bedrock with little to thin soil cover. Any PCBs that may have been released from former transformers would have been transported via runoff and overland flow across the bedrock outcrop of Round Island to the Hudson River. While sediment and soil was noted along the northeastern edge of DU-24 off the bedrock outcrop of Round Island, this sediment/soil was along the shoreline of the tidally influenced Hudson River. The Hudson River itself is a PCB source. Therefore, collecting PCBs from soil/sediment along the Hudson River would not provide information that could be used to evaluate a potential release related to former transformers.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 7b DU-23 and DU-25 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	DU-23 (T1 Northwest of Building 406)							DU-25 (T3 Northeast of Building 220)							
			Sample ID	IINY-DU-23A-SSIS-20200320		IINY-DU-23B-SSIS-20200320		IINY-DU-23C-SSIS-20200320		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-DU-25A-SSIS-20200320		IINY-DU-25B-SSIS-20200320		IINY-DU-25C-SSIS-20200320		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
			Sample Date	3/20/2020 9:15		3/20/2020 9:15		3/20/2020 9:15				3/20/2020 10:30		3/20/2020 10:30		3/20/2020 10:30			
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**																
Soil pH																			
pH	N/A	N/A	N/A	6.0		5.4		5.5		5.71	6.44	6.2		6.4		6.4		1.82	6.62
Polychlorinated Biphenyls																			
PCB, Total	1336-36-3	0.041	mg/kg	0.034	J	0.025	J	0.019	J	29.000	0.045	0.011	J	0.016	J	0.012	J	20.400	0.020
PCB-1016 (Aroclor 1016)	12674-11-2	0.041	mg/kg	0.03	U (LOD)	0.03	U (LOD)	0.03	U (LOD)	ND	ND	0.03	U (LOD)	0.03	U (LOD)	0.029	U (LOD)	ND	ND
PCB-1221 (Aroclor 1221)	11104-28-2	0.041	mg/kg	0.077	U (LOD)	0.077	U (LOD)	0.079	U (LOD)	ND	ND	0.078	U (LOD)	0.077	U (LOD)	0.076	U (LOD)	ND	ND
PCB-1232 (Aroclor 1232)	11141-16-5	0.041	mg/kg	0.03	U (LOD)	0.03	U (LOD)	0.03	U (LOD)	ND	ND	0.03	U (LOD)	0.03	U (LOD)	0.029	U (LOD)	ND	ND
PCB-1242 (Aroclor 1242)	53469-21-9	0.041	mg/kg	0.046	U (LOD)	0.046	U (LOD)	0.047	U (LOD)	ND	ND	0.047	U (LOD)	0.046	U (LOD)	0.046	U (LOD)	ND	ND
PCB-1248 (Aroclor 1248)	12672-29-6	0.041	mg/kg	0.015	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND	0.015	U (LOD)	0.014	U (LOD)	0.014	U (LOD)	ND	ND
PCB-1254 (Aroclor 1254)	11097-69-1	0.041	mg/kg	0.032	U (LOD)	0.032	U (LOD)	0.033	U (LOD)	ND	ND	0.032	U (LOD)	0.032	U (LOD)	0.032	U (LOD)	ND	ND
PCB-1260 (Aroclor 1260)	11096-82-5	0.041	mg/kg	0.034	J	0.025	J	0.019	J	29.000	0.045	0.011	J	0.016	J	0.012	J	20.400	0.020
PCB-1262 (Aroclor 1262)	37324-23-5	0.041	mg/kg	0.015	U (LOD)	0.015	U (LOD)	0.016	U (LOD)	ND	ND	0.016	U (LOD)	0.015	U (LOD)	0.015	U (LOD)	ND	ND
PCB-1268 (Aroclor 1268)	11100-14-4	0.041	mg/kg	0.0065	U (LOD)	0.0065	U (LOD)	0.0066	U (LOD)	ND	ND	0.0065	U (LOD)	0.0064	U (LOD)	0.0063	U (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

DU-24 (T2 near AOC #16 – Area Dumping Area) was not sampled. The majority of DU-24 contained exposed bedrock with little to thin soil cover. Any PCBs that may have been released from former transformers would have been transported via runoff and overland flow across the bedrock outcrop of Round Island to the Hudson River. While sediment and soil was noted along the northeastern edge of DU-24 off the bedrock outcrop of Round Island, this sediment/soil was along the shoreline of the tidally influenced Hudson River. The Hudson River itself is a PCB source. Therefore, collecting PCBs from soil/sediment along the Hudson River would not provide information that could be used to evaluate a potential release related to former transformers.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 8a EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	EXDU-1 (Building 609 – Mine Loading Plant)							EXDU-2 (Building 506 – Filling House #3)							EXDU-3 (Building 503 – Filling House #6)						
				Sample ID	IINY-EXDU-1A-SSIS-20200317		IINY-EXDU-1B-SSIS-20200317		IINY-EXDU-1C-SSIS-20200317		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-2A-SSIS-20200317		IINY-EXDU-2B-SSIS-20200317		IINY-EXDU-2C-SSIS-20200317		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-3A-SSIS-20200318		IINY-EXDU-3B-SSIS-20200318		IINY-EXDU-3C-SSIS-20200318		% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6				0-6		0-6		0-6			
				Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
				Sample Date	3/17/2020 15:35		3/17/2020 15:35		3/17/2020 15:35				3/17/2020 16:10		3/17/2020 16:10		3/17/2020 16:10				3/18/2020 8:00		3/18/2020 8:00		3/18/2020 8:00			
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit																								
Soil pH																												
pH	N/A	N/A	N/A	N/A	7.0		6.9		6.9		0.833	7.08	7.0		6.8		6.9		1.5	7.2	6.3		6.5		5.5		8.7	7.4
Explosives Residues																												
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.029	J	16.9	0.0513	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.038	J	0.039	J	0.051	J	17	0.0609	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.28	J	0.21	J	0.37	J	28	0.489	0.099	UJ (LOQ)	0.1	UJ (LOQ)	0.1	UJ (LOQ)	ND	ND	0.093	UJ (LOQ)	0.097	UJ (LOQ)	0.096	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.063	J	0.06	J	0.22		80	0.345	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.097	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.099	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.093	U (LOQ)	0.097	U (LOQ)	0.096	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.019	U (LOD)	0.02	U (LOD)	0.02	U (LOD)	ND	ND	0.02	U (LOD)	0.02	U (LOD)	0.02	U (LOD)	ND	ND	0.019	U (LOD)	0.019	U (LOD)	0.019	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.19	U (LOQ)	0.2	U (LOQ)	0.2	U (LOQ)	ND	ND	0.2	U (LOQ)	0.2	U (LOQ)	0.2	U (LOQ)	ND	ND	0.19	U (LOQ)	0.19	U (LOQ)	0.19	U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.097	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.099	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.093	U (LOQ)	0.097	U (LOQ)	0.096	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.19	U (LOD)	0.2	U (LOD)	0.2	U (LOD)	ND	ND	0.2	U (LOD)	0.2	U (LOD)	0.2	U (LOD)	ND	ND	0.19	U (LOD)	0.19	U (LOD)	0.19	U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.68	U (LOD)	0.7	U (LOD)	0.7	U (LOD)	ND	ND	0.69	U (LOD)	0.72	U (LOD)	0.71	U (LOD)	ND	ND	0.65	U (LOD)	0.68	U (LOD)	0.67	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	390	5,700	mg/kg	0.068	U (LOD)	0.07	U (LOD)	0.07	U (LOD)	ND	ND	0.069	U (LOD)	0.072	U (LOD)	0.071	U (LOD)	ND	ND	0.065	U (LOD)	0.068	U (LOD)	0.067	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.97	U (LOD)	1	U (LOD)	1	U (LOD)	ND	ND	0.99	U (LOD)	1	U (LOD)	1	U (LOD)	ND	ND	0.93	U (LOD)	0.97	U (LOD)	0.96	U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.16	230	mg/kg	0.097	UJ (LOD)	0.1	UJ (LOD)	0.1	UJ (LOD)	ND	ND	0.099	UJ (LOD)	0.1	UJ (LOD)	0.1	UJ (LOD)	ND	ND	0.093	UJ (LOD)	0.097	UJ (LOD)	0.096	UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 8a EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	EXDU-4 (Building 404 – Filling House #1)							EXDU-5 (Building 307 – Filling House #5)						
				Sample ID	IINY-EXDU-4A-SSIS-20200317	IINY-EXDU-4B-SSIS-20200317	IINY-EXDU-4C-SSIS-20200317	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-5A-SSIS-20200318	IINY-EXDU-5B-SSIS-20200318	IINY-EXDU-5C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/17/2020 14:30	3/17/2020 14:30	3/17/2020 14:30			3/18/2020 9:30	3/18/2020 9:30	3/18/2020 9:30		
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**										
Soil pH														
pH	N/A	N/A	N/A	N/A	6.5	6.3	6.8	3.85	7.17	7.1	7.2	7.3	1.39	7.45
Explosives Residues														
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.039 U (LOD)	0.04 U (LOD)	0.04 U (LOD)	ND	ND	0.039 U (LOD)	0.037 U (LOD)	0.037 U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.039 U (LOD)	0.04 U (LOD)	0.04 U (LOD)	ND	ND	0.039 U (LOD)	0.037 U (LOD)	0.037 U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.098 UJ (LOQ)	0.1 UJ (LOQ)	0.1 UJ (LOQ)	ND	ND	0.097 UJ (LOQ)	0.093 UJ (LOQ)	0.092 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.039 U (LOD)	0.04 U (LOD)	0.04 U (LOD)	ND	ND	0.039 U (LOD)	0.037 U (LOD)	0.037 U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.039 U (LOD)	0.04 U (LOD)	0.04 U (LOD)	ND	ND	0.039 U (LOD)	0.037 U (LOD)	0.037 U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.098 U (LOQ)	0.1 U (LOQ)	0.1 U (LOQ)	ND	ND	0.097 U (LOQ)	0.093 U (LOQ)	0.092 U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.098 U (LOD)	0.1 U (LOD)	0.1 U (LOD)	ND	ND	0.097 U (LOD)	0.093 U (LOD)	0.092 U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.02 UJ (LOD)	0.02 UJ (LOD)	0.02 UJ (LOD)	ND	ND	0.019 U (LOD)	0.019 U (LOD)	0.018 U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.2 U (LOQ)	0.2 U (LOQ)	0.2 U (LOQ)	ND	ND	0.19 U (LOQ)	0.19 U (LOQ)	0.18 U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.098 U (LOQ)	0.1 U (LOQ)	0.1 U (LOQ)	ND	ND	0.097 U (LOQ)	0.093 U (LOQ)	0.092 U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.098 U (LOD)	0.1 U (LOD)	0.1 U (LOD)	ND	ND	0.097 U (LOD)	0.093 U (LOD)	0.092 U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.098 U (LOD)	0.1 U (LOD)	0.1 U (LOD)	ND	ND	0.097 U (LOD)	0.093 U (LOD)	0.092 U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.2 U (LOD)	0.2 U (LOD)	0.2 U (LOD)	ND	ND	0.19 U (LOD)	0.19 U (LOD)	0.18 U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.69 U (LOD)	0.7 U (LOD)	0.71 U (LOD)	ND	ND	0.68 U (LOD)	0.65 U (LOD)	0.65 U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	390	5,700	mg/kg	0.069 U (LOD)	0.07 U (LOD)	0.071 U (LOD)	ND	ND	0.068 U (LOD)	0.065 U (LOD)	0.065 U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.98 U (LOD)	1 U (LOD)	1 U (LOD)	ND	ND	0.97 U (LOD)	0.93 U (LOD)	0.92 U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.16	230	mg/kg	0.098 UJ (LOD)	0.1 UJ (LOD)	0.1 UJ (LOD)	ND	ND	0.097 UJ (LOD)	0.093 UJ (LOD)	0.092 UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

Table 8a EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	EXDU-6 (Building 306 – Filling House #2)							EXDU-7 (Building 202 – Shell House #5 and D Plant)						
				Sample ID	IINY-EXDU-6A-SSIS-20200318	IINY-EXDU-6B-SSIS-20200318	IINY-EXDU-6C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-7A-SSIS-20200318	IINY-EXDU-7B-SSIS-20200318	IINY-EXDU-7C-SSIS-20200318	% RSD	95% Upper Confidence Limit Chebychev Test
				Depth (in)	0-6	0-6	0-6			0-6	0-6	0-6		
				Matrix	Surface Soil	Surface Soil	Surface Soil			Surface Soil	Surface Soil	Surface Soil		
				Sample Date	3/18/2020 10:15	3/18/2020 10:15	3/18/2020 10:15			3/18/2020 11:45	3/18/2020 11:45	3/18/2020 11:45		
Analyte	CAS #	Residential Soil RSL (mg/kg)[a,b]	Industrial Soil RSL (mg/kg)[a]	Unit										
Soil pH														
pH	N/A	N/A	N/A	N/A	7.2	7.3	7.3	0.795	7.41	7.0	7.0	7.1	0.821	7.18
Explosives Residues														
1,3,5-Trinitrobenzene (TNB)	99-35-4	220	3,200	mg/kg	0.038 U (LOD)	0.039 U (LOD)	0.039 U (LOD)	ND	ND	0.038 U (LOD)	0.039 U (LOD)	0.037 U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.63	8.2	mg/kg	0.038 U (LOD)	0.039 U (LOD)	0.039 U (LOD)	ND	ND	0.038 U (LOD)	0.039 U (LOD)	0.037 U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	3.6	51	mg/kg	0.094 UJ (LOQ)	0.096 UJ (LOQ)	0.099 UJ (LOQ)	ND	ND	0.094 UJ (LOQ)	0.099 UJ (LOQ)	0.093 UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	1.7	7.4	mg/kg	0.038 U (LOD)	0.039 U (LOD)	0.039 U (LOD)	ND	ND	0.038 U (LOD)	0.039 U (LOD)	0.037 U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	0.36	1.5	mg/kg	0.038 U (LOD)	0.039 U (LOD)	0.039 U (LOD)	ND	ND	0.038 U (LOD)	0.039 U (LOD)	0.037 U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	0.77	11	mg/kg	0.094 U (LOQ)	0.096 U (LOQ)	0.099 U (LOQ)	ND	ND	0.094 U (LOQ)	0.099 U (LOQ)	0.093 U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	3.2	15	mg/kg	0.094 U (LOD)	0.096 U (LOD)	0.099 U (LOD)	ND	ND	0.094 U (LOD)	0.099 U (LOD)	0.093 U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	NS	mg/kg	0.019 U (LOD)	0.019 U (LOD)	0.02 U (LOD)	ND	ND	0.019 U (LOD)	0.02 U (LOD)	0.019 U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.63	8.2	mg/kg	0.19 U (LOQ)	0.19 U (LOQ)	0.2 U (LOQ)	ND	ND	0.19 U (LOQ)	0.2 U (LOQ)	0.19 U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	0.77	11	mg/kg	0.094 U (LOQ)	0.096 U (LOQ)	0.099 U (LOQ)	ND	ND	0.094 U (LOQ)	0.099 U (LOQ)	0.093 U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	25	140	mg/kg	0.094 U (LOD)	0.096 U (LOD)	0.099 U (LOD)	ND	ND	0.094 U (LOD)	0.099 U (LOD)	0.093 U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	8.3	38	mg/kg	0.094 U (LOD)	0.096 U (LOD)	0.099 U (LOD)	ND	ND	0.094 U (LOD)	0.099 U (LOD)	0.093 U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	5.1	22	mg/kg	0.19 U (LOD)	0.19 U (LOD)	0.2 U (LOD)	ND	ND	0.19 U (LOD)	0.2 U (LOD)	0.19 U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	0.63	8.2	mg/kg	0.66 U (LOD)	0.67 U (LOD)	0.69 U (LOD)	ND	ND	0.66 U (LOD)	0.69 U (LOD)	0.65 U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	390	5,700	mg/kg	0.066 U (LOD)	0.067 U (LOD)	0.069 U (LOD)	ND	ND	0.066 U (LOD)	0.069 U (LOD)	0.065 U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	13	160	mg/kg	0.94 U (LOD)	0.96 U (LOD)	0.99 U (LOD)	ND	ND	0.94 U (LOD)	0.99 U (LOD)	0.93 U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.16	230	mg/kg	0.094 UJ (LOD)	0.096 UJ (LOD)	0.099 UJ (LOD)	ND	ND	0.094 UJ (LOD)	0.099 UJ (LOD)	0.093 UJ (LOD)	ND	ND

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
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N/A = not applicable
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RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 8b EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	EXDU-1 (Building 609 – Mine Loading Plant)								EXDU-2 (Building 506 – Filling House #3)								EXDU-3 (Building 503 – Filling House #6)							
			Sample ID	IINY-EXDU-1A-SSIS-20200317		IINY-EXDU-1B-SSIS-20200317		IINY-EXDU-1C-SSIS-20200317		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-2A-SSIS-20200317		IINY-EXDU-2B-SSIS-20200317		IINY-EXDU-2C-SSIS-20200317		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-3A-SSIS-20200318		IINY-EXDU-3B-SSIS-20200318		IINY-EXDU-3C-SSIS-20200318		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6				0-6		0-6		0-6			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
			Sample Date	3/17/2020 15:35		3/17/2020 15:35		3/17/2020 15:35				3/17/2020 16:10		3/17/2020 16:10		3/17/2020 16:10				3/18/2020 8:00		3/18/2020 8:00		3/18/2020 8:00			
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit																								
Soil pH																											
pH	N/A	N/A	N/A	7.0		6.9		6.9		0.833	7.08	7.0		6.8		6.9		1.5	7.2	6.3		6.5		5.5		8.7	7.4
Explosives Residues																											
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.029	J	16.9	0.0513	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.038	J	0.039	J	0.051	J	17	0.0609	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.28	J	0.21	J	0.37	J	28	0.489	0.099	UJ (LOQ)	0.1	UJ (LOQ)	0.1	UJ (LOQ)	ND	ND	0.093	UJ (LOQ)	0.097	UJ (LOQ)	0.096	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.063	J	0.06	J	0.22		80	0.345	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.04	U (LOD)	0.041	U (LOD)	0.04	U (LOD)	ND	ND	0.037	U (LOD)	0.039	U (LOD)	0.038	U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.097	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.099	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.093	U (LOQ)	0.097	U (LOQ)	0.096	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.019	U (LOD)	0.02	U (LOD)	0.02	U (LOD)	ND	ND	0.02	U (LOD)	0.02	U (LOD)	0.02	U (LOD)	ND	ND	0.019	U (LOD)	0.019	U (LOD)	0.019	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.19	U (LOQ)	0.2	U (LOQ)	0.2	U (LOQ)	ND	ND	0.2	U (LOQ)	0.2	U (LOQ)	0.2	U (LOQ)	ND	ND	0.19	U (LOQ)	0.19	U (LOQ)	0.19	U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.097	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.099	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.093	U (LOQ)	0.097	U (LOQ)	0.096	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.097	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.099	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.093	U (LOD)	0.097	U (LOD)	0.096	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.19	U (LOD)	0.2	U (LOD)	0.2	U (LOD)	ND	ND	0.2	U (LOD)	0.2	U (LOD)	0.2	U (LOD)	ND	ND	0.19	U (LOD)	0.19	U (LOD)	0.19	U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.68	U (LOD)	0.7	U (LOD)	0.7	U (LOD)	ND	ND	0.69	U (LOD)	0.72	U (LOD)	0.71	U (LOD)	ND	ND	0.65	U (LOD)	0.68	U (LOD)	0.67	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	16	mg/kg	0.068	U (LOD)	0.07	U (LOD)	0.07	U (LOD)	ND	ND	0.069	U (LOD)	0.072	U (LOD)	0.071	U (LOD)	ND	ND	0.065	U (LOD)	0.068	U (LOD)	0.067	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.97	U (LOD)	1	U (LOD)	1	U (LOD)	ND	ND	0.99	U (LOD)	1	U (LOD)	1	U (LOD)	ND	ND	0.93	U (LOD)	0.97	U (LOD)	0.96	U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.018	mg/kg	0.097	UJ (LOD)	0.1	UJ (LOD)	0.1	UJ (LOD)	ND	ND	0.099	UJ (LOD)	0.1	UJ (LOD)	0.1	UJ (LOD)	ND	ND	0.093	UJ (LOD)	0.097	UJ (LOD)	0.096	UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 8b EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	EXDU-4 (Building 404 – Filling House #1)							EXDU-5 (Building 307 – Filling House #5)						
			Sample ID	IINY-EXDU-4A-SSIS-20200317		IINY-EXDU-4B-SSIS-20200317		IINY-EXDU-4C-SSIS-20200317		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-5A-SSIS-20200318		IINY-EXDU-5B-SSIS-20200318		IINY-EXDU-5C-SSIS-20200318		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
			Sample Date	3/17/2020 14:30		3/17/2020 14:30		3/17/2020 14:30				3/18/2020 9:30		3/18/2020 9:30		3/18/2020 9:30			
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**																
Soil pH																			
pH	N/A	N/A	N/A	6.5		6.3		6.8		3.85	7.17	7.1		7.2		7.3		1.39	7.45
Explosives Residues																			
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.039	U (LOD)	0.037	U (LOD)	0.037	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.039	U (LOD)	0.037	U (LOD)	0.037	U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.098	UJ (LOQ)	0.1	UJ (LOQ)	0.1	UJ (LOQ)	ND	ND	0.097	UJ (LOQ)	0.093	UJ (LOQ)	0.092	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.039	U (LOD)	0.037	U (LOD)	0.037	U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.039	U (LOD)	0.04	U (LOD)	0.04	U (LOD)	ND	ND	0.039	U (LOD)	0.037	U (LOD)	0.037	U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.098	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.097	U (LOQ)	0.093	U (LOQ)	0.092	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.098	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.097	U (LOD)	0.093	U (LOD)	0.092	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.02	UJ (LOD)	0.02	UJ (LOD)	0.02	UJ (LOD)	ND	ND	0.019	U (LOD)	0.019	U (LOD)	0.018	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.2	U (LOQ)	0.2	U (LOQ)	0.2	U (LOQ)	ND	ND	0.19	U (LOQ)	0.19	U (LOQ)	0.18	U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.098	U (LOQ)	0.1	U (LOQ)	0.1	U (LOQ)	ND	ND	0.097	U (LOQ)	0.093	U (LOQ)	0.092	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.098	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.097	U (LOD)	0.093	U (LOD)	0.092	U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.098	U (LOD)	0.1	U (LOD)	0.1	U (LOD)	ND	ND	0.097	U (LOD)	0.093	U (LOD)	0.092	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.2	U (LOD)	0.2	U (LOD)	0.2	U (LOD)	ND	ND	0.19	U (LOD)	0.19	U (LOD)	0.18	U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.69	U (LOD)	0.7	U (LOD)	0.71	U (LOD)	ND	ND	0.68	U (LOD)	0.65	U (LOD)	0.65	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	16	mg/kg	0.069	U (LOD)	0.07	U (LOD)	0.071	U (LOD)	ND	ND	0.068	U (LOD)	0.065	U (LOD)	0.065	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.98	U (LOD)	1	U (LOD)	1	U (LOD)	ND	ND	0.97	U (LOD)	0.93	U (LOD)	0.92	U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.018	mg/kg	0.098	UJ (LOD)	0.1	UJ (LOD)	0.1	UJ (LOD)	ND	ND	0.097	UJ (LOD)	0.093	UJ (LOD)	0.092	UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

Table 8b EXDU-1 Through EXDU-7 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	EXDU-6 (Building 306 – Filling House #2)							EXDU-7 (Building 202 – Shell House #5 and D Plant)						
			Sample ID	IINY-EXDU-6A-SSIS-20200318		IINY-EXDU-6B-SSIS-20200318		IINY-EXDU-6C-SSIS-20200318		% RSD	95% Upper Confidence Limit Chebychev Test	IINY-EXDU-7A-SSIS-20200318		IINY-EXDU-7B-SSIS-20200318		IINY-EXDU-7C-SSIS-20200318		% RSD	95% Upper Confidence Limit Chebychev Test
			Depth (in)	0-6		0-6		0-6				0-6		0-6		0-6			
			Matrix	Surface Soil		Surface Soil		Surface Soil				Surface Soil		Surface Soil		Surface Soil			
			Sample Date	3/18/2020 10:15		3/18/2020 10:15		3/18/2020 10:15				3/18/2020 11:45		3/18/2020 11:45		3/18/2020 11:45			
Analyte	CAS #	EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]	Unit																
Soil pH																			
pH	N/A	N/A	N/A	7.2		7.3		7.3		0.795	7.41	7.0		7.0		7.1		0.821	7.18
Explosives Residues																			
1,3,5-Trinitrobenzene (TNB)	99-35-4	0.3	mg/kg	0.038	U (LOD)	0.039	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.039	U (LOD)	0.037	U (LOD)	ND	ND
1,3-Dinitrobenzene (DNB)	99-65-0	0.034	mg/kg	0.038	U (LOD)	0.039	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.039	U (LOD)	0.037	U (LOD)	ND	ND
2,4,6-Trinitrotoluene (TNT)	118-96-7	7.5	mg/kg	0.094	UJ (LOQ)	0.096	UJ (LOQ)	0.099	UJ (LOQ)	ND	ND	0.094	UJ (LOQ)	0.099	UJ (LOQ)	0.093	UJ (LOQ)	ND	ND
2,4-Dinitrotoluene (24DNT)	121-14-2	6	mg/kg	0.038	U (LOD)	0.039	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.039	U (LOD)	0.037	U (LOD)	ND	ND
2,6-Dinitrotoluene (26DNT)	606-20-2	4	mg/kg	0.038	U (LOD)	0.039	U (LOD)	0.039	U (LOD)	ND	ND	0.038	U (LOD)	0.039	U (LOD)	0.037	U (LOD)	ND	ND
2-Amino-4,6-dinitrotoluene (2AMDNT)	35572-78-2	14	mg/kg	0.094	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND	0.094	U (LOQ)	0.099	U (LOQ)	0.093	U (LOQ)	ND	ND
2-Nitrotoluene (2NT)	88-72-2	0.19	mg/kg	0.094	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND	0.094	U (LOD)	0.099	U (LOD)	0.093	U (LOD)	ND	ND
3,5-Dinitroaniline	618-87-1	NS	mg/kg	0.019	U (LOD)	0.019	U (LOD)	0.02	U (LOD)	ND	ND	0.019	U (LOD)	0.02	U (LOD)	0.019	U (LOD)	ND	ND
3-Nitrotoluene (3NT)	99-08-1	0.13	mg/kg	0.19	U (LOQ)	0.19	U (LOQ)	0.2	U (LOQ)	ND	ND	0.19	U (LOQ)	0.2	U (LOQ)	0.19	U (LOQ)	ND	ND
4-Amino-2,6-dinitrotoluene (4AMDNT)	19406-51-0	12	mg/kg	0.094	U (LOQ)	0.096	U (LOQ)	0.099	U (LOQ)	ND	ND	0.094	U (LOQ)	0.099	U (LOQ)	0.093	U (LOQ)	ND	ND
4-Nitrotoluene (4NT)	99-99-0	0.14	mg/kg	0.094	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND	0.094	U (LOD)	0.099	U (LOD)	0.093	U (LOD)	ND	ND
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	2.3	mg/kg	0.094	U (LOD)	0.096	U (LOD)	0.099	U (LOD)	ND	ND	0.094	U (LOD)	0.099	U (LOD)	0.093	U (LOD)	ND	ND
Nitrobenzene (NB)	98-95-3	2.2	mg/kg	0.19	U (LOD)	0.19	U (LOD)	0.2	U (LOD)	ND	ND	0.19	U (LOD)	0.2	U (LOD)	0.19	U (LOD)	ND	ND
Nitroglycerin (NG)	55-63-0	13	mg/kg	0.66	U (LOD)	0.67	U (LOD)	0.69	U (LOD)	ND	ND	0.66	U (LOD)	0.69	U (LOD)	0.65	U (LOD)	ND	ND
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	16	mg/kg	0.066	U (LOD)	0.067	U (LOD)	0.069	U (LOD)	ND	ND	0.066	U (LOD)	0.069	U (LOD)	0.065	U (LOD)	ND	ND
Pentaerythritol tetranitrate (PETN)	78-11-5	2.2	mg/kg	0.94	U (LOD)	0.96	U (LOD)	0.99	U (LOD)	ND	ND	0.94	U (LOD)	0.99	U (LOD)	0.93	U (LOD)	ND	ND
N-Methyl-2,4,6-trinitrophenylnitramine	479-45-8	0.018	mg/kg	0.094	UJ (LOD)	0.096	UJ (LOD)	0.099	UJ (LOD)	ND	ND	0.094	UJ (LOD)	0.099	UJ (LOD)	0.093	UJ (LOD)	ND	ND

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
ND = not detected
NS = No standard
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 9a BADU-1 Through BADU-8 Phase I Incremental Soil Sampling Analytical Results Compared to Human Health Screening Criteria

				Location	BADU-1	BADU-2	BADU-3	BADU-4	BADU-5	BADU-6	BADU-7	BADU-8	95% Upper Confidence Limit Chebychev Test
				Sample ID	IINY-BADU-1	IINY-BADU-2	IINY-BADU-3	IINY-BADU-4	IINY-BADU-5	IINY-BADU-6	IINY-BADU-7	IINY-BADU-8	
				Depth (in)	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	
				Matrix	Surface Soil	Surface Soil	Surface Soil	Surface Soil	Surface Soil	Surface Soil	Surface Soil	Surface Soil	
				Sample Date / Time	4/1/2020 11:45	4/1/2020 12:15	4/1/2020 12:45	4/1/2020 13:20	4/1/2020 9:55	4/1/2020 9:20	4/1/2020 9:00	4/1/2020 9:25	
Analyte	**CAS #**	**Residential Soil RSL (mg/kg)[a,b]**	**Industrial Soil RSL (mg/kg)[a]**	**Unit**									
Soil pH													
pH	N/A	N/A	N/A	N/A	6.3	6.7	6	6.1	6.1	5.9	6.1	6	6.54
Mercury													
Mercury	7439-97-6	1.1	4.6	mg/kg	0.290	0.310	0.260	0.200	0.120	0.320	0.200	0.120	0.351
Target Analyte List Metals													
Aluminum	7429-90-5	7,700	110,000	mg/kg	14,000	15,000	11,000	7,300	10,000	11,000	15,000	14,000	16,400
Antimony	7440-36-0	3.1	47	mg/kg	1.2	1.2	1.1	0.94	0.53	1.2	0.89	0.83	1.35
Arsenic	7440-38-2	0.68	3	mg/kg	4.3	4	3.5	2.1	6.3	7.5	7.1	7.8	8.59
Barium	7440-39-3	1,500	22,000	mg/kg	28	21	17	13	47	47	47	47	56.7
Beryllium	7440-41-7	16	230	mg/kg	0.31	0.23	0.11	0.057 J	0.92	0.7	0.62	0.57	0.914
Cadmium	7440-43-9	7.1	98	mg/kg	0.22	0.14	0.13	0.11	0.23	0.29	0.16	0.12	0.274
Calcium	7440-70-2	NS	NS	mg/kg	2,100	900	1,300	980	2,000	1,100	750	650	2,070
Chromium, Total	7440-47-3	12,000	180,000	mg/kg	7.4	6.4	6.9	3.6	22 J	20	20	31	29.9
Cobalt	7440-48-4	2.3	35	mg/kg	2.1	1.7	1.1	0.59	6.4	5.3	5.5	7.3	7.83
Copper	7440-50-8	310	4,700	mg/kg	11 J	8.5 J	7 J	4.9 J	25	21 J	16 J	26	27.7
Iron	7439-89-6	5,500	82,000	mg/kg	12,000	13,000	13,000	8,600	14,000	15,000	17,000	21,000	19,800
Lead	7439-92-1	400	800	mg/kg	53	57	50	33	49	94	58	46	82.1
Magnesium	7439-95-4	NS	NS	mg/kg	1,400	1,300	1,000	600	2,100 J	1,900	2,300	3,100	2,940
Manganese	7439-96-5	180	2,600	mg/kg	330	200	75	37	340	200	230	280	381
Nickel	7440-02-0	150	2,200	mg/kg	5.7	4.8	4	2.7	22	23	19	23	27.6
Potassium	2023695	NS	NS	mg/kg	840	710	840	700	620	720	670	720	846
Selenium	7782-49-2	39	580	mg/kg	0.91	0.92	0.9	0.65	1.1	1.2	1.1	0.9	1.22
Silver	7440-22-4	39	580	mg/kg	0.33	0.28	0.4	0.28	0.14	0.33	0.25	0.17	0.405
Sodium	7440-23-5	NS	NS	mg/kg	52 J	38 J	34 J	36 J	63 J	44 J	36 J	47 J	59.1
Thallium	7440-28-0	0.078	1.2	mg/kg	0.1	0.099	0.082 J	0.043 J	0.2	0.18	0.17	0.16	0.215
Vanadium	7440-62-2	39	580	mg/kg	23	21	22	13	37 J	40	47	56	55.3
Zinc	7440-66-6	2,300	35,000	mg/kg	35	28	21 J	30	60	72	45	48	69.1
Polycyclic Aromatic Hydrocarbons													
1-Methylnaphthalene	90-12-0	18	73	mg/kg	0.028 J	0.015 J	0.012 J	0.0099 J	0.018 J	0.015 J	0.0053 J	0.0039 J	0.0252
2-Methylnaphthalene	91-57-6	24	300	mg/kg	0.022 J	0.015 J	0.013 J	0.011 J	0.017 J	0.012 J	0.0052 J	0.0039 J	0.0215
Acenaphthene	83-32-9	360	4,500	mg/kg	0.097 J	0.034 J	0.024 J	0.021 J	0.049 J	0.072 J	0.0081 J	0.0048 J	0.0883
Acenaphthylene	208-96-8	NS	NS	mg/kg	0.05 J	0.034 J	0.028 J	0.025 J	0.081 J	0.028 J	0.013 J	0.009 J	0.0688
Anthracene	120-12-7	1,800	23,000	mg/kg	0.12 J	0.049 J	0.039 J	0.028 J	0.12 J	0.094 J	0.015 J	0.0084 J	0.13
Benzo(a)anthracene	56-55-3	1.1	21	mg/kg	0.68 J	0.21 J	0.17 J	0.1 J	0.51 J	0.68 J	0.067 J	0.043 J	0.727
Benzo(a)pyrene	50-32-8	0.11	2.1	mg/kg	0.79 J	0.22 J	0.19 J	0.12 J	0.52 J	0.69 J	0.063 J	0.045 J	0.783
Benzo(b)fluoranthene	205-99-2	1.1	21	mg/kg	1.3 J	0.38 J	0.31 J	0.19 J	0.73 J	1.1 J	0.11 J	0.072 J	1.24
Benzo(g,h,i)perylene	191-24-2	NS	NS	mg/kg	0.68 J	0.18 J	0.15 J	0.091 J	0.39 J	0.52 J	0.049 J	0.035 J	0.632
Benzo(k)fluoranthene	207-08-9	11	210	mg/kg	0.46 J	0.14 J	0.11 J	0.074 J	0.27 J	0.41 J	0.039 J	0.028 J	0.451
Chrysene	218-01-9	110	2,100	mg/kg	1 J	0.31 J	0.25 J	0.16 J	0.62 J	0.9 J	0.093 J	0.061 J	0.992
Dibenz(a,h)anthracene	53-70-3	0.11	2.1	mg/kg	0.15 J	0.042 J	0.036 J	0.027 R (LOD)	0.092 J	0.13 J	0.013 J	0.0063 UJ (LOD)	0.147
Fluoranthene	206-44-0	240	3,000	mg/kg	2.2 J	0.7 J	0.54 J	0.31 J	1.3 J	1.9 J	0.19 J	0.12 J	2.14
Fluorene	86-73-7	240	3,000	mg/kg	0.1 J	0.087 J	0.056 J	0.06 J	0.065 J	0.061 J	0.01 J	0.0058 J	0.107
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	21	mg/kg	0.74 J	0.2 J	0.17 J	0.1 J	0.42 J	0.58 J	0.053 J	0.037 J	0.692
Naphthalene	91-20-3	2	8.6	mg/kg	0.031 J	0.023 J	0.017 J	0.017 J	0.022 J	0.017 J	0.0074 J	0.0063 J	0.0301
Phenanthrene	85-01-8	NS	NS	mg/kg	1.1 J	0.36 J	0.28 J	0.17 J	0.58 J	0.86 J	0.09 J	0.061 J	1.02
Pyrene	129-00-0	180	2,300	mg/kg	1.9 J	0.6 J	0.46 J	0.27 J	1.1 J	1.5 J	0.16 J	0.11 J	1.79
Total LMW PAHs	N/A	NS	NS	mg/kg	1.548	0.617	0.469	0.3419	0.952	1.159	0.154	0.1031	1.46
Total HMW PAHs	N/A	NS	NS	mg/kg	9.9	2.982	2.386	1.415	5.952	8.41	0.837	0.551	9.59

(a) EPA Regional Screening Levels Table, EPA, November 2019. Value shown is equal to HI=0.1. Carcinogenic values equal to $1x10^{-6}$.
(b) A deed restriction is in place noting that there is to be no residential development at the site (USACE 1995); however, EPA residential soil RSLs are included to assess for children receptors including site visitation for nature walks and potential camping.

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
X = Usability Pending Review
Red concentrations exceed EPA Residential Soil RSL.
Concentrations highlighted in yellow exceed EPA Industrial Soil RSL.

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Table 9b BADU-1 Through BADU-8 Phase I Incremental Soil Sampling Analytical Results Compared to Ecological Screening Criteria

			Location	BADU-1		BADU-2		BADU-3		BADU-4		BADU-5		BADU-6		BADU-7		BADU-8		95% Upper Confidence Limit Chebychev Test
			Sample ID	IINY-BADU-1		IINY-BADU-2		IINY-BADU-3		IINY-BADU-4		IINY-BADU-5		IINY-BADU-6		IINY-BADU-7		IINY-BADU-8		
			Depth (in)	0-6		0-6		0-6		0-6		0-6		0-6		0-6		0-6		
			Matrix	Surface Soil		Surface Soil		Surface Soil		Surface Soil		Surface Soil		Surface Soil		Surface Soil		Surface Soil		
			Sample Date / Time	4/1/2020 11:45		4/1/2020 12:15		4/1/2020 12:45		4/1/2020 13:20		4/1/2020 9:55		4/1/2020 9:20		4/1/2020 9:00		4/1/2020 9:25		
Analyte	**CAS #**	**EPA Region 4 Ecological Soil Screening Levels (mg/kg)[a]**	**Unit**																	
Soil pH																				
pH	N/A	N/A	N/A	6.3		6.7		6		6.1		6.1		5.9		6.1		6		6.54
Mercury																				
Mercury	7439-97-6	0.013	mg/kg	0.290		0.310		0.260		0.200		0.120		0.320		0.200		0.120		0.351
Target Analyte List Metals																				
Aluminum	7429-90-5	NS	mg/kg	14,000		15,000		11,000		7,300		10,000		11,000		15,000		14,000		16,400
Antimony	7440-36-0	0.27	mg/kg	1.2		1.2		1.1		0.94		0.53		1.2		0.89		0.83		1.35
Arsenic	7440-38-2	6.8	mg/kg	4.3		4		3.5		2.1		6.3		7.5		7.1		7.8		8.59
Barium	7440-39-3	110	mg/kg	28		21		17		13		47		47		47		47		56.7
Beryllium	7440-41-7	2.5	mg/kg	0.31		0.23		0.11		0.057	J	0.92		0.7		0.62		0.57		0.914
Cadmium	7440-43-9	0.36	mg/kg	0.22		0.14		0.13		0.11		0.23		0.29		0.16		0.12		0.274
Calcium	7440-70-2	NS	mg/kg	2,100		900		1,300		980		2,000		1,100		750		650		2,070
Chromium, Total	7440-47-3	23	mg/kg	7.4		6.4		6.9		3.6		22	J	20		20		31		29.9
Cobalt	7440-48-4	13	mg/kg	2.1		1.7		1.1		0.59		6.4		5.3		5.5		7.3		7.83
Copper	7440-50-8	28	mg/kg	11	J	8.5	J	7	J	4.9	J	25		21	J	16	J	26		27.7
Iron	7439-89-6	NS	mg/kg	12,000		13,000		13,000		8,600		14,000		15,000		17,000		21,000		19,800
Lead	7439-92-1	11	mg/kg	53		57		50		33		49		94		58		46		82.1
Magnesium	7439-95-4	NS	mg/kg	1,400		1,300		1,000		600		2,100	J	1,900		2,300		3,100		2,940
Manganese	7439-96-5	220	mg/kg	330		200		75		37		340		200		230		280		381
Nickel	7440-02-0	38	mg/kg	5.7		4.8		4		2.7		22		23		19		23		27.6
Potassium	2023695	NS	mg/kg	840		710		840		700		620		720		670		720		846
Selenium	7782-49-2	0.52	mg/kg	0.91		0.92		0.9		0.65		1.1		1.2		1.1		0.9		1.22
Silver	7440-22-4	4.2	mg/kg	0.33		0.28		0.4		0.28		0.14		0.33		0.25		0.17		0.405
Sodium	7440-23-5	NS	mg/kg	52	J	38	J	34	J	36	J	63	J	44	J	36	J	47	J	59.1
Thallium	7440-28-0	0.05	mg/kg	0.1		0.099		0.082	J	0.043	J	0.2		0.18		0.17		0.16		0.215
Vanadium	7440-62-2	7.8	mg/kg	23		21		22		13		37	J	40		47		56		55.3
Zinc	7440-66-6	46	mg/kg	35		28		21	J	30		60		72		45		48		69.1
Polycyclic Aromatic Hydrocarbons																				
1-Methylnaphthalene	90-12-0	29	mg/kg	0.028	J	0.015	J	0.012	J	0.0099	J	0.018	J	0.015	J	0.0053	J	0.0039	J	0.0252
2-Methylnaphthalene	91-57-6	29	mg/kg	0.022	J	0.015	J	0.013	J	0.011	J	0.017	J	0.012	J	0.0052	J	0.0039	J	0.0215
Acenaphthene	83-32-9	29	mg/kg	0.097	J	0.034	J	0.024	J	0.021	J	0.049	J	0.072	J	0.0081	J	0.0048	J	0.0883
Acenaphthylene	208-96-8	29	mg/kg	0.05	J	0.034	J	0.028	J	0.025	J	0.081	J	0.028	J	0.013	J	0.009	J	0.0688
Anthracene	120-12-7	29	mg/kg	0.12	J	0.049	J	0.039	J	0.028	J	0.12	J	0.094	J	0.015	J	0.0084	J	0.13
Benzo(a)anthracene	56-55-3	1.1	mg/kg	0.68	J	0.21	J	0.17	J	0.1	J	0.51	J	0.68	J	0.067	J	0.043	J	0.727
Benzo(a)pyrene	50-32-8	1.1	mg/kg	0.79	J	0.22	J	0.19	J	0.12	J	0.52	J	0.69	J	0.063	J	0.045	J	0.783
Benzo(b)fluoranthene	205-99-2	1.1	mg/kg	1.3	J	0.38	J	0.31	J	0.19	J	0.73	J	1.1	J	0.11	J	0.072	J	1.24
Benzo(g,h,i)perylene	191-24-2	1.1	mg/kg	0.68	J	0.18	J	0.15	J	0.091	J	0.39	J	0.52	J	0.049	J	0.035	J	0.632
Benzo(k)fluoranthene	207-08-9	1.1	mg/kg	0.46	J	0.14	J	0.11	J	0.074	J	0.27	J	0.41	J	0.039	J	0.028	J	0.451
Chrysene	218-01-9	1.1	mg/kg	1	J	0.31	J	0.25	J	0.16	J	0.62	J	0.9	J	0.093	J	0.061	J	0.992
Dibenz(a,h)anthracene	53-70-3	1.1	mg/kg	0.15	J	0.042	J	0.036	J	0.027	R (LOD)	0.092	J	0.13	J	0.013	J	0.0063	UJ (LOD)	0.147
Fluoranthene	206-44-0	1.1	mg/kg	2.2	J	0.7	J	0.54	J	0.31	J	1.3	J	1.9	J	0.19	J	0.12	J	2.14
Fluorene	86-73-7	29	mg/kg	0.1	J	0.087	J	0.056	J	0.06	J	0.065	J	0.061	J	0.01	J	0.0058	J	0.107
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	mg/kg	0.74	J	0.2	J	0.17	J	0.1	J	0.42	J	0.58	J	0.053	J	0.037	J	0.692
Naphthalene	91-20-3	29	mg/kg	0.031	J	0.023	J	0.017	J	0.017	J	0.022	J	0.017	J	0.0074	J	0.0063	J	0.0301
Phenanthrene	85-01-8	29	mg/kg	1.1	J	0.36	J	0.28	J	0.17	J	0.58	J	0.86	J	0.09	J	0.061	J	1.02
Pyrene	129-00-0	1.1	mg/kg	1.9	J	0.6	J	0.46	J	0.27	J	1.1	J	1.5	J	0.16	J	0.11	J	1.79
Total LMW PAHs	N/A	29	mg/kg	1.548		0.617		0.469		0.3419		0.952		1.159		0.154		0.1031		1.46
Total HMW PAHs	N/A	1.1	mg/kg	9.9		2.982		2.386		1.415		5.952		8.41		0.837		0.551		9.59

(a) EPA Region 4 Ecological Risk Assessment Supplemental Guidance. Scientific Support Section Superfund Division EPA Region 4. March 2018. Screening values are further detailed in Appendix E, Ecological Risk Assessment Work Plan

NOTE:
mg/kg = milligrams per kilogram
CAS = Chemical Abstracts Service
EPA = United States Environmental Protection Agency
J = Estimated: The analyte was positively identified; the quantitation is an estimation
LOD = Limit of detection
LOQ = Limit of quantitation
N/A = not applicable
NS = No standard
PCB = Polychlorinated biphenyl
RSL = regional screening level
U = Analyte not detected. Numerical value is LOD or LOQ, as indicated.
UJ = Analyte not detected, and associated numerical value (LOD or LOQ) is approximate.
X = Usability Pending Review
Concentrations highlighted in blue exceed EPA Region 4 Ecological Soil Screening Levels.

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Table 10 Constituents Exceeding Levels at Each Decision Unit (Based on 95% UCLs)

Decision Unit	Soil Type	Constituent Exceeding Screening Criteria[1,2]		
		Ecological Soil Screening Level	EPA Residential Regional Screening Level	EPA Industrial Regional Screening Level
DU-1	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Lead, Manganese, Thallium, Vanadium, Zinc, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene	Arsenic
DU-2	Surface Soil	Mercury, Antimony, Arsenic, Lead, Manganese, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene , Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene, Benzo(a)anthracene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Arsenic, Benzo(a)pyrene
DU-3	Surface Soil	Mercury, Antimony, Arsenic, Copper, Lead, Manganese, Thallium, Vanadium, Zinc, Benzo(b)fluoranthene, Fluoranthene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene	Arsenic
DU-4	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Total Chromium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Lead, Manganese, Thallium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Arsenic
DU-5	Surface Soil	Mercury, Antimony, Arsenic, Lead, Manganese, Thallium, Vanadium, Zinc, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene	Arsenic
DU-6	Surface Soil	Antimony, Arsenic, Copper, Lead, Manganese, Thallium	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium	Arsenic
DU-7	Surface Soil	Antimony, Arsenic, Barium, Cadmium, Copper, Lead, Manganese, Thallium, Vanadium, Zinc	Aluminum, Antimony, Arsenic, Cobalt, Iron, Lead, Manganese, Thallium	Arsenic, Lead
DU-9	Surface Soil	Mercury, Antimony, Arsenic, Copper , Lead, Manganese, Thallium, Vanadium, Zinc, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene	Arsenic
DU-10	Surface Soil	Antimony, Arsenic, Cadmium, Lead, Manganese, Thallium, Vanadium, Zinc	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium	Arsenic
DU-11	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic
DU-12	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene , Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)pyrene

Table 10 Constituents Exceeding Levels at Each Decision Unit (Based on 95% UCLs)

Decision Unit	Soil Type	Constituent Exceeding Screening Criteria[1,2]		
		Ecological Soil Screening Level	EPA Residential Regional Screening Level	EPA Industrial Regional Screening Level
DU-13	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Antimony, Arsenic, Cobalt, Copper, Iron, Manganese, Thallium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene
DU-14	Surface Soil	Mercury, Antimony, Arsenic, Copper, Lead, Manganese, Thallium, Vanadium, Zinc	Aluminum, Arsenic, Cobalt, Copper, Iron, Manganese, Thallium	Arsenic
DU-15	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium	Arsenic
DU-16	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Thallium, Vanadium, Zinc	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium	Arsenic
DU-17	Surface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Total Chromium, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(b)fluoranthene, Chrysene, Fluoranthene, Pyrene, Total HMW PAHs, Total PCBs, PCB-1248 (Aroclor 1248) , PCB-1260 (Aroclor 1260)	Mercury, Aluminum, Antimony, Arsenic, Cadmium, Cobalt, Copper, Iron, Lead, Manganese, Thallium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene	Arsenic, Lead
	Subsurface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Copper, Lead, Selenium, Thallium, Vanadium, Zinc, Benzo(b)fluoranthene, Chrysene, Fluoranthene, Pyrene, Total HMW PAHs	Antimony, Arsenic, Cobalt, Iron, Lead, Thallium, Zinc, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene	Arsenic, Lead
DU-18	Surface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Total Chromium, Copper, Lead, Manganese, Nickel, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Chrysene, Fluoranthene, Pyrene, Total HMW PAHs, Total PCBs, PCB-1248 (Aroclor 1248), PCB-1260 (Aroclor 1260)	Mercury, Aluminum, Antimony, Arsenic, Cobalt, Copper, Iron, Lead, Manganese, Thallium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Total PCBs, PCB-1260 (Aroclor 1260)	Mercury, Arsenic, Lead
	Subsurface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Total Chromium, Copper, Lead, Manganese, Nickel, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(b)fluoranthene, Chrysene, Fluoranthene, Pyrene, Total HMW PAHs, Total PCBs, PCB-1262 (Aroclor 1262)	Mercury, Antimony, Arsenic, Cadmium, Cobalt, Copper, Iron, Lead, Manganese, Thallium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene	Copper, Arsenic, Lead

Table 10 Constituents Exceeding Levels at Each Decision Unit (Based on 95% UCLs)

Decision Unit	Soil Type	Constituent Exceeding Screening Criteria[1,2]		
		Ecological Soil Screening Level	EPA Residential Regional Screening Level	EPA Industrial Regional Screening Level
DU-19	Surface Soil	Mercury, Antimony, Arsenic, Cadmium, Cobalt, Copper, Lead, Manganese, Selenium, Thallium, Vanadium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Vanadium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)pyrene
	Subsurface Soil	Mercury, Cobalt, Copper, Lead, Manganese, Thallium, Vanadium, Zinc, Total PCBs, PCB-1260 (Aroclor 1260)	Aluminum, Arsenic, Cobalt, Iron, Manganese, Thallium, Vanadium	Arsenic
DU-20	Surface Soil	Total HMW PAHs	Benzo(a)pyrene	No Exceedances
	Subsurface Soil	No Exceedances	No Exceedances	No Exceedances
DU-21	Surface Soil	Total HMW PAHs	Benzo(a)pyrene	No Exceedances
	Subsurface Soil	No Exceedances	No Exceedances	No Exceedances
DU-22	Surface Soil	Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene , Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Benzo(a)pyrene
DU-23	Surface Soil	Total PCBs, PCB-1260 (Aroclor 1260)	No Exceedances	No Exceedances
DU-25	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-1	Surface Soil	1,3-Dinitrobenzene (DNB)	No Exceedances	No Exceedances
EXDU-2	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-3	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-4	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-5	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-6	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-7	Surface Soil	No Exceedances	No Exceedances	No Exceedances

NOTES:
1. United States Environmental Protection Agency (EPA) Regional Screening Levels (RSLs) Table, EPA, May 2020.
2. EPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008.
Note:
DU = Decision Unit
HMW = High molecular weight
LMW = Low molecular weight
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl

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Table 11 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-01	3	3	0.004608154	8	8	0.013412476	-0.008804321	0.96	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-02	3	3	0.052322388	8	8	0.013412476	0.038909912	<0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-03	3	3	0.010696411	8	8	0.013412476	-0.002716064	0.67	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-04	3	2	0.041000366	8	8	0.013412476	0.027587891	0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-05	3	3	0.007843018	8	8	0.013412476	-0.005569458	0.88	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-09	3	3	0.00630188	8	8	0.013412476	-0.007110596	0.94	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-11	3	3	0.026000977	8	8	0.013412476	0.012588501	0.02	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-12	3	3	0.056671143	8	8	0.013412476	0.043258667	<0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-13	3	3	0.363327026	8	8	0.013412476	0.349914551	0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-16	3	3	0.020568848	8	8	0.013412476	0.007156372	0.13	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-17	3	3	0.031005859	8	8	0.013412476	0.017593384	0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-18	3	3	0.040328979	8	8	0.013412476	0.026916504	<0.01	Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-19	3	3	0.017333984	8	8	0.013412476	0.003921509	0.23	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-20	3	3	0.008728027	8	8	0.013412476	-0.004684448	0.84	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-21	3	3	0.005599976	8	8	0.013412476	-0.0078125	0.96	Do Not Reject H0
SS	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-22	3	3	0.040664673	8	8	0.013412476	0.027252197	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-01	3	3	0.007095337	8	8	0.012390137	-0.0052948	0.93	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-02	3	3	0.042007446	8	8	0.012390137	0.02961731	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-03	3	3	0.012268066	8	8	0.012390137	-0.00012207	0.55	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-04	3	2	0.054000854	8	8	0.012390137	0.041610718	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-05	3	3	0.010131836	8	8	0.012390137	-0.002258301	0.72	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-09	3	3	0.008651733	8	8	0.012390137	-0.003738403	0.84	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-11	3	3	0.041000366	8	8	0.012390137	0.028610229	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-12	3	3	0.068664551	8	8	0.012390137	0.056274414	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-13	3	3	0.413330078	8	8	0.012390137	0.400939941	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-16	3	3	0.027404785	8	8	0.012390137	0.015014648	0.05	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-17	3	3	0.046340942	8	8	0.012390137	0.033950806	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-18	3	3	0.067337036	8	8	0.012390137	0.054946899	0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-19	3	3	0.027008057	8	8	0.012390137	0.01461792	<0.01	Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-20	3	3	0.013000488	8	8	0.012390137	0.000610352	0.40	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-21	3	3	0.007659912	8	8	0.012390137	-0.004730225	0.90	Do Not Reject H0
SS	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-22	3	3	0.061340332	8	8	0.012390137	0.048950195	<0.01	Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-01	3	3	0.006393433	8	8	0.038757324	-0.032363892	0.97	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-02	3	3	0.17666626	8	8	0.038757324	0.137908936	0.01	Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-03	3	3	0.02633667	8	8	0.038757324	-0.012420654	0.68	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-04	3	3	0.101654053	8	8	0.038757324	0.062896729	0.09	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-05	3	3	0.016326904	8	8	0.038757324	-0.02243042	0.86	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-09	3	3	0.010604858	8	8	0.038757324	-0.028152466	0.94	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-11	3	3	0.021347046	8	8	0.038757324	-0.017410278	0.78	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-12	3	3	0.430007935	8	8	0.038757324	0.39125061	<0.01	Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-13	3	3	3.933334351	8	8	0.038757324	3.894577026	<0.01	Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-16	3	3	0.108657837	8	8	0.038757324	0.069900513	0.02	Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-17	3	3	0.035675049	8	8	0.038757324	-0.003082275	0.54	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-18	3	3	0.050994873	8	8	0.038757324	0.012237549	0.28	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-19	3	3	0.037658691	8	8	0.038757324	-0.001098633	0.50	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-20	3	3	0.011444092	8	8	0.038757324	-0.027313232	0.94	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-21	3	3	0.011672974	8	8	0.038757324	-0.027084351	0.89	Do Not Reject H0
SS	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-22	3	3	0.095657349	8	8	0.038757324	0.056900024	<0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-01	3	3	0.031326294	8	8	0.033493042	-0.002166748	0.52	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-02	3	3	0.11000061	8	8	0.033493042	0.076507568	<0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-03	3	3	0.039657593	8	8	0.033493042	0.006164551	0.31	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-04	3	3	0.209991455	8	8	0.033493042	0.176498413	<0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-05	3	3	0.019668579	8	8	0.033493042	-0.013824463	0.83	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-09	3	3	0.030670166	8	8	0.033493042	-0.002822876	0.45	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-11	3	3	0.186676025	8	8	0.033493042	0.153182983	0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-12	3	3	0.059661865	8	8	0.033493042	0.026168823	0.06	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-13	3	1	0.566665649	8	8	0.033493042	0.533172607	<0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-16	3	3	0.031341553	8	8	0.033493042	-0.002151489	0.48	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-17	3	3	0.13999939	8	8	0.033493042	0.106506348	0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-18	3	3	0.084335327	8	8	0.033493042	0.050842285	0.03	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-19	3	3	0.203338623	8	8	0.033493042	0.169845581	<0.01	Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-20	3	3	0.034011841	8	8	0.033493042	0.000518799	0.47	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-21	3	3	0.022659302	8	8	0.033493042	-0.01083374	0.76	Do Not Reject H0
SS	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-22	3	3	0.196670532	8	8	0.033493042	0.16317749	<0.01	Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-01	3	3	11000	8	8	12162.5	-1162.5	0.76	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-02	3	3	13000	8	8	12162.5	837.5	0.40	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-03	3	3	13000	8	8	12162.5	837.5	0.38	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-04	3	3	10000	8	8	12162.5	-2162.5	0.87	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-05	3	3	13000	8	8	12162.5	837.5	0.41	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-06	3	3	10666.66667	8	8	12162.5	-1495.833328	0.83	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-07	3	3	10666.66667	8	8	12162.5	-1495.833328	0.87	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-09	3	3	10666.66667	8	8	12162.5	-1495.833328	0.87	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-10	3	3	10433.33333	8	8	12162.5	-1729.166672	0.89	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-11	3	3	10233.33333	8	8	12162.5	-1929.166672	0.91	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-12	3	3	11000	8	8	12162.5	-1162.5	0.76	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-13	3	3	11333.33333	8	8	12162.5	-829.1666718	0.68	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-14	3	3	10333.33333	8	8	12162.5	-1829.166672	0.91	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-15	3	3	11000	8	8	12162.5	-1162.5	0.78	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-16	3	3	14000	8	8	12162.5	1837.5	0.21	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-17	3	3	5900	8	8	12162.5	-6262.5	1.00	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-18	3	3	9500	8	8	12162.5	-2662.5	0.91	Do Not Reject H0
SS	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-19	3	3	20333.33334	8	8	12162.5	8170.833344	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-01	3	3	0.028320313	8	8	0.059188843	-0.03086853	0.83	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-02	3	3	0.476669312	8	8	0.059188843	0.417480469	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-03	3	3	0.091339111	8	8	0.059188843	0.032150269	0.17	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-04	3	3	0.313323975	8	8	0.059188843	0.254135132	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-05	3	3	0.060668945	8	8	0.059188843	0.001480103	0.52	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-09	3	3	0.03666687	8	8	0.059188843	-0.022521973	0.70	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-11	3	3	0.125671387	8	8	0.059188843	0.066482544	0.03	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-12	3	3	0.733337402	8	8	0.059188843	0.67414856	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-13	3	3	6.166656494	8	8	0.059188843	6.107467651	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-16	3	3	0.134338379	8	8	0.059188843	0.075149536	0.03	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-17	3	3	0.186660767	8	8	0.059188843	0.127471924	0.02	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-18	3	3	0.169998169	8	8	0.059188843	0.110809326	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-19	3	3	0.28666687	8	8	0.059188843	0.227478027	<0.01	Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-20	3	3	0.04133606	8	8	0.059188843	-0.017852783	0.73	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-21	3	3	0.040664673	8	8	0.059188843	-0.01852417	0.72	Do Not Reject H0
SS	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-22	3	3	0.333328247	8	8	0.059188843	0.274139404	<0.01	Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-01	3	3	0.5	8	8	0.986236572	-0.486236572	1.00	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-02	3	3	0.636672974	8	8	0.986236572	-0.349563599	0.98	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-03	3	3	0.659988403	8	8	0.986236572	-0.326248169	0.98	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-04	3	3	1.699996948	8	8	0.986236572	0.713760376	0.01	Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-05	3	3	0.516662598	8	8	0.986236572	-0.469573975	1.00	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-06	3	3	0.866653442	8	8	0.986236572	-0.11958313	0.76	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-07	3	3	1.616668701	8	8	0.986236572	0.630432129	0.07	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-09	3	3	0.433334351	8	8	0.986236572	-0.552902222	1.00	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-10	3	3	0.38999939	8	8	0.986236572	-0.596237183	1.00	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-11	3	3	0.82333374	8	8	0.986236572	-0.162902832	0.86	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-12	3	3	0.636672974	8	8	0.986236572	-0.349563599	0.98	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-13	3	3	1.699996948	8	8	0.986236572	0.713760376	0.02	Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-14	3	3	0.583343506	8	8	0.986236572	-0.402893066	0.98	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-15	3	3	0.833328247	8	8	0.986236572	-0.152908325	0.83	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-16	3	3	0.419998169	8	8	0.986236572	-0.566238403	1.00	Do Not Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-17	3	3	7.900009155	8	8	0.986236572	6.913772583	<0.01	Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-18	3	3	6.533325195	8	8	0.986236572	5.547088623	<0.01	Reject H0
SS	Antimony	SB	7440-36-0	mg/kg	IINY-DU-19	3	3	0.42666626	8	8	0.986236572	-0.559570313	1.00	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-01	3	3	10.6333313	8	8	5.325012207	5.308319092	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-02	3	3	13.66667175	8	8	5.325012207	8.341659546	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-03	3	3	13.33334351	8	8	5.325012207	8.008331299	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-04	3	3	31.33334351	8	8	5.325012207	26.0083313	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-05	3	3	11.33332825	8	8	5.325012207	6.00831604	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-06	3	3	9.666656494	8	8	5.325012207	4.341644287	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-07	3	3	17.33332825	8	8	5.325012207	12.00831604	0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-09	3	3	7.066665649	8	8	5.325012207	1.741653442	0.14	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-10	3	3	7.066665649	8	8	5.325012207	1.741653442	0.15	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-11	3	3	39.66665649	8	8	5.325012207	34.34164429	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-12	3	3	13.66667175	8	8	5.325012207	8.341659546	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-13	3	3	10.56666565	8	8	5.325012207	5.241653442	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-14	3	3	17	8	8	5.325012207	11.67498779	<0.01	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-15	3	3	6.866668701	8	8	5.325012207	1.541656494	0.16	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-16	3	3	7.033325195	8	8	5.325012207	1.708312988	0.13	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-17	3	3	6.566665649	8	8	5.325012207	1.241653442	0.20	Do Not Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-18	3	3	12.6000061	8	8	5.325012207	7.274993896	0.02	Reject H0
SS	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-19	3	3	9.466674805	8	8	5.325012207	4.141662598	0.03	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-01	3	3	67.33334351	8	8	33.375	33.95834351	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-10	3	3	57	8	8	33.375	23.625	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-02	3	3	73.66667175	8	8	33.375	40.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-03	3	3	79.66667175	8	8	33.375	46.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-04	3	3	85	8	8	33.375	51.625	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-05	3	3	72.66667175	8	8	33.375	39.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-06	3	3	65.66667175	8	8	33.375	32.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-07	3	3	104.3333282	8	8	33.375	70.95832825	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-09	3	3	61.66665649	8	8	33.375	28.29165649	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-11	3	3	77.66665649	8	8	33.375	44.29165649	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-12	3	3	80.66667175	8	8	33.375	47.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-13	3	3	90	8	8	33.375	56.625	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-14	3	3	78.33332825	8	8	33.375	44.95832825	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-15	3	3	60.33332825	8	8	33.375	26.95832825	0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-16	3	3	58.66667175	8	8	33.375	25.29167175	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-17	3	3	133.3333435	8	8	33.375	99.95834351	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-18	3	3	276.6666718	8	8	33.375	243.2916718	<0.01	Reject H0
SS	Barium	BA	7440-39-3	mg/kg	IINY-DU-19	3	3	75.66667175	8	8	33.375	42.29167175	<0.01	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-01	3	3	0.130004883	8	8	0.307479858	-0.177474976	0.76	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-02	3	3	2.066665649	8	8	0.307479858	1.759185791	<0.01	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-03	3	3	0.38999939	8	8	0.307479858	0.082519531	0.34	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-04	3	3	1.130004883	8	8	0.307479858	0.822525024	<0.01	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-05	3	3	0.316665649	8	8	0.307479858	0.009185791	0.51	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-09	3	3	0.156661987	8	8	0.307479858	-0.150817871	0.72	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-11	3	3	0.720001221	8	8	0.307479858	0.412521362	0.03	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-12	3	3	1.933334351	8	8	0.307479858	1.625854492	<0.01	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-13	3	3	14.1333313	8	8	0.307479858	13.82585144	0.02	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-16	3	3	0.383346558	8	8	0.307479858	0.075866699	0.38	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-17	3	3	0.649993896	8	8	0.307479858	0.342514038	0.05	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-18	3	3	0.706680298	8	8	0.307479858	0.399200439	0.07	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-19	3	3	1.566665649	8	8	0.307479858	1.259185791	<0.01	Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-20	3	3	0.14666748	8	8	0.307479858	-0.160812378	0.73	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-21	3	3	0.148666382	8	8	0.307479858	-0.158813477	0.80	Do Not Reject H0
SS	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-22	3	3	1.5	8	8	0.307479858	1.192520142	0.02	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-01	3	3	0.149993896	8	8	0.329742432	-0.179748535	0.79	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-02	3	3	1.733322144	8	8	0.329742432	1.403579712	<0.01	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-03	3	3	0.403335571	8	8	0.329742432	0.07359314	0.37	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-04	3	3	1.100006104	8	8	0.329742432	0.770263672	<0.01	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-05	3	3	0.279998779	8	8	0.329742432	-0.049743652	0.59	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-09	3	3	0.17666626	8	8	0.329742432	-0.153076172	0.77	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-11	3	3	0.940002441	8	8	0.329742432	0.61026001	0.02	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-12	3	3	1.600006104	8	8	0.329742432	1.270263672	<0.01	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-13	3	3	10.69999695	8	8	0.329742432	10.37025452	<0.01	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-16	3	3	0.380004883	8	8	0.329742432	0.050262451	0.38	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-17	3	3	0.553329468	8	8	0.329742432	0.223587036	0.17	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-18	3	3	0.64666748	8	8	0.329742432	0.316925049	0.04	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-19	3	3	1.259994507	8	8	0.329742432	0.930252075	<0.01	Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-20	3	3	0.159988403	8	8	0.329742432	-0.169754028	0.76	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-21	3	3	0.13633728	8	8	0.329742432	-0.193405151	0.81	Do Not Reject H0
SS	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-22	3	3	1.433334351	8	8	0.329742432	1.103591919	<0.01	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-01	3	3	0.233337402	8	8	0.523986816	-0.290649414	0.76	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-02	3	3	2.466659546	8	8	0.523986816	1.942672729	0.02	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-03	3	3	0.593322754	8	8	0.523986816	0.069335938	0.42	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-04	3	3	2.566665649	8	8	0.523986816	2.042678833	0.01	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-05	3	3	0.410003662	8	8	0.523986816	-0.113983154	0.61	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-09	3	3	0.279998779	8	8	0.523986816	-0.243988037	0.74	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-11	3	3	1.300003052	8	8	0.523986816	0.776016235	0.02	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-12	3	3	2.333343506	8	8	0.523986816	1.809356689	<0.01	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-13	3	3	14.56666565	8	8	0.523986816	14.04267883	0.02	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-16	3	3	0.563339233	8	8	0.523986816	0.039352417	0.46	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-17	3	3	0.96333313	8	8	0.523986816	0.439346313	0.09	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-18	3	3	1.186676025	8	8	0.523986816	0.662689209	0.04	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-19	3	3	2.266662598	8	8	0.523986816	1.742675781	<0.01	Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-20	3	3	0.253326416	8	8	0.523986816	-0.2706604	0.76	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-21	3	3	0.226669312	8	8	0.523986816	-0.297317505	0.76	Do Not Reject H0
SS	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-22	3	3	2.266677856	8	8	0.523986816	1.74269104	<0.01	Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-01	3	3	0.116668701	8	8	0.261871338	-0.145202637	0.80	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-02	3	3	0.976669312	8	8	0.261871338	0.714797974	0.01	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-03	3	3	0.289993286	8	8	0.261871338	0.028121948	0.36	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-04	3	3	0.880004883	8	8	0.261871338	0.618133545	<0.01	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-05	3	3	0.179992676	8	8	0.261871338	-0.081878662	0.67	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-09	3	3	0.120010376	8	8	0.261871338	-0.141860962	0.77	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-11	3	3	0.933334351	8	8	0.261871338	0.671463013	<0.01	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-12	3	3	1.129989624	8	8	0.261871338	0.868118286	0.01	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-13	3	3	6.433334351	8	8	0.261871338	6.171463013	<0.01	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-16	3	3	0.25	8	8	0.261871338	-0.011871338	0.53	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-17	3	3	0.35333252	8	8	0.261871338	0.091461182	0.23	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-18	3	3	0.449996948	8	8	0.261871338	0.18812561	0.14	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-19	3	3	0.763336182	8	8	0.261871338	0.501464844	0.03	Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-20	3	3	0.123336792	8	8	0.261871338	-0.138534546	0.76	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-21	3	3	0.080337524	8	8	0.261871338	-0.181533813	0.88	Do Not Reject H0
SS	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-22	3	3	0.806671143	8	8	0.261871338	0.544799805	0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-01	3	3	0.077667236	8	8	0.191375732	-0.113708496	0.86	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-02	3	3	0.763336182	8	8	0.191375732	0.571960449	<0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-03	3	3	0.186676025	8	8	0.191375732	-0.004699707	0.49	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-04	3	3	0.803329468	8	8	0.191375732	0.611953735	<0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-05	3	3	0.133331299	8	8	0.191375732	-0.058044434	0.66	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-09	3	3	0.094665527	8	8	0.191375732	-0.096710205	0.76	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-11	3	3	0.453338623	8	8	0.191375732	0.261962891	0.03	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-12	3	3	0.89666748	8	8	0.191375732	0.705291748	<0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-13	3	3	5.866653442	8	8	0.191375732	5.67527771	<0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-16	3	3	0.203323364	8	8	0.191375732	0.011947632	0.41	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-17	3	3	0.300003052	8	8	0.191375732	0.108627319	0.17	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-18	3	3	0.32333374	8	8	0.191375732	0.131958008	0.12	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-19	3	3	0.793334961	8	8	0.191375732	0.601959229	<0.01	Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-20	3	3	0.082000732	8	8	0.191375732	-0.109375	0.78	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-21	3	3	0.075668335	8	8	0.191375732	-0.115707397	0.83	Do Not Reject H0
SS	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-22	3	3	0.759994507	8	8	0.191375732	0.568618774	<0.01	Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-01	3	3	0.523330688	8	8	0.43963623	0.083694458	0.36	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-02	3	3	0.579986572	8	8	0.43963623	0.140350342	0.27	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-03	3	3	0.556671143	8	8	0.43963623	0.117034912	0.27	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-04	3	3	0.483337402	8	8	0.43963623	0.043701172	0.46	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-05	3	3	0.580001831	8	8	0.43963623	0.140365601	0.23	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-06	3	3	0.529998779	8	8	0.43963623	0.090362549	0.36	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-07	3	3	0.53666687	8	8	0.43963623	0.09703064	0.34	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-09	3	3	0.496658325	8	8	0.43963623	0.057022095	0.44	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-10	3	3	0.553329468	8	8	0.43963623	0.113693237	0.29	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-11	3	3	0.550003052	8	8	0.43963623	0.110366821	0.29	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-12	3	3	0.64666748	8	8	0.43963623	0.20703125	0.18	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-13	3	3	0.599990845	8	8	0.43963623	0.160354614	0.24	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-14	3	3	0.550003052	8	8	0.43963623	0.110366821	0.30	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-15	3	3	0.479995728	8	8	0.43963623	0.040359497	0.44	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-16	3	3	0.576675415	8	8	0.43963623	0.137039185	0.29	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-17	3	3	0.35333252	8	8	0.43963623	-0.086303711	0.68	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-18	3	3	0.590011597	8	8	0.43963623	0.150375366	0.25	Do Not Reject H0
SS	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-19	3	3	0.406661987	8	8	0.43963623	-0.032974243	0.58	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-01	3	3	0.283325195	8	8	0.174987793	0.108337402	0.02	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-02	3	3	0.226669312	8	8	0.174987793	0.051681519	0.13	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-03	3	3	0.253326416	8	8	0.174987793	0.078338623	0.06	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-04	3	3	0.986679077	8	8	0.174987793	0.811691284	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-05	3	3	0.209991455	8	8	0.174987793	0.035003662	0.22	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-06	3	0	1.7	8	8	0.174987793	1.525012207	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-07	3	3	0.563323975	8	8	0.174987793	0.388336182	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-09	3	3	0.236663818	8	8	0.174987793	0.061676025	0.12	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-10	3	2	0.179992676	8	8	0.174987793	0.005004883	0.44	Do Not Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-11	3	3	0.366668701	8	8	0.174987793	0.191680908	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-12	3	3	0.35333252	8	8	0.174987793	0.178344727	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-13	3	3	0.753341675	8	8	0.174987793	0.578353882	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-14	3	3	0.276672363	8	8	0.174987793	0.10168457	0.02	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-15	3	3	0.513336182	8	8	0.174987793	0.338348389	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-16	3	3	0.283340454	8	8	0.174987793	0.108352661	0.02	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-17	3	3	13.66665649	8	8	0.174987793	13.4916687	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-18	3	3	4.633331299	8	8	0.174987793	4.458343506	<0.01	Reject H0
SS	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-19	3	3	0.293334961	8	8	0.174987793	0.118347168	0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-01	3	3	4533.333328	8	8	1222.5	3310.833328	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-02	3	3	10600	8	8	1222.5	9377.5	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-03	3	3	6233.333344	8	8	1222.5	5010.833344	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-04	3	3	9666.666656	8	8	1222.5	8444.166656	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-05	3	3	3233.333344	8	8	1222.5	2010.833344	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-06	3	3	14333.33333	8	8	1222.5	13110.83333	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-07	3	3	16000	8	8	1222.5	14777.5	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-09	3	3	7366.666672	8	8	1222.5	6144.166672	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-10	3	3	15000	8	8	1222.5	13777.5	0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-11	3	3	26666.66666	8	8	1222.5	25444.16666	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-12	3	3	11333.33334	8	8	1222.5	10110.83334	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-13	3	3	8866.666672	8	8	1222.5	7644.166672	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-14	3	3	8400	8	8	1222.5	7177.5	0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-15	3	3	8166.666672	8	8	1222.5	6944.166672	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-16	3	3	7600	8	8	1222.5	6377.5	0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-17	3	3	58333.33333	8	8	1222.5	57110.83333	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-18	3	3	12333.33333	8	8	1222.5	11110.83333	<0.01	Reject H0
SS	Calcium	CA	7440-70-2	mg/kg	IINY-DU-19	3	3	12333.33333	8	8	1222.5	11110.83333	<0.01	Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-01	3	3	13.33332825	8	8	14.6625061	-1.329177856	0.57	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-02	3	3	15	8	8	14.6625061	0.337493896	0.44	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-03	3	3	15.33332825	8	8	14.6625061	0.670822144	0.43	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-04	3	3	17	8	8	14.6625061	2.337493896	0.36	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-05	3	3	15	8	8	14.6625061	0.337493896	0.45	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-06	3	3	13	8	8	14.6625061	-1.662506104	0.62	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-07	3	3	16	8	8	14.6625061	1.337493896	0.40	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-09	3	3	13	8	8	14.6625061	-1.662506104	0.66	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-10	3	3	13.66667175	8	8	14.6625061	-0.995834351	0.55	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-11	3	3	15	8	8	14.6625061	0.337493896	0.45	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-12	3	3	15	8	8	14.6625061	0.337493896	0.45	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-13	3	3	14.66667175	8	8	14.6625061	0.004165649	0.46	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-14	3	3	13.66667175	8	8	14.6625061	-0.995834351	0.58	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-15	3	3	15	8	8	14.6625061	0.337493896	0.42	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-16	3	3	14.66667175	8	8	14.6625061	0.004165649	0.49	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-17	3	3	16.96665955	8	8	14.6625061	2.304153442	0.39	Do Not Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-18	3	3	95.66667175	8	8	14.6625061	81.00416565	0.04	Reject H0
SS	Chromium	CR	7440-47-3	mg/kg	IINY-DU-19	3	3	10.33332825	8	8	14.6625061	-4.329177856	0.80	Do Not Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-01	3	3	0.143325806	8	8	0.424224854	-0.280899048	0.89	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-02	3	3	1.966674805	8	8	0.424224854	1.542449951	<0.01	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-03	3	3	0.403335571	8	8	0.424224854	-0.020889282	0.51	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-04	3	3	1.833328247	8	8	0.424224854	1.409103394	0.03	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-05	3	3	0.313339233	8	8	0.424224854	-0.11088562	0.64	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-09	3	3	0.17666626	8	8	0.424224854	-0.247558594	0.81	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-11	3	3	1.003326416	8	8	0.424224854	0.579101563	0.02	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-12	3	3	2.033325195	8	8	0.424224854	1.609100342	<0.01	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-13	3	3	13.7666626	8	8	0.424224854	13.34243774	<0.01	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-16	3	3	0.380004883	8	8	0.424224854	-0.044219971	0.58	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-17	3	3	0.743331909	8	8	0.424224854	0.319107056	0.08	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-18	3	3	1.153335571	8	8	0.424224854	0.729110718	0.02	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-19	3	3	1.459991455	8	8	0.424224854	1.035766602	0.03	Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-20	3	3	0.173339844	8	8	0.424224854	-0.25088501	0.83	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-21	3	3	0.152328491	8	8	0.424224854	-0.271896362	0.88	Do Not Reject H0
SS	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-22	3	3	1.599990845	8	8	0.424224854	1.175765991	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-01	3	3	7.100006104	8	8	3.748733521	3.351272583	0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-02	3	3	7.566665649	8	8	3.748733521	3.817932129	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-03	3	3	7.633331299	8	8	3.748733521	3.884597778	0.02	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-04	3	3	7.300003052	8	8	3.748733521	3.551269531	0.02	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-05	3	3	7.799987793	8	8	3.748733521	4.051254272	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-06	3	3	7.233337402	8	8	3.748733521	3.484603882	0.02	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-07	3	3	6.100006104	8	8	3.748733521	2.351272583	0.13	Do Not Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-09	3	3	7.433334351	8	8	3.748733521	3.68460083	0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-10	3	3	7.800003052	8	8	3.748733521	4.051269531	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-11	3	3	6.800003052	8	8	3.748733521	3.051269531	0.03	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-12	3	3	7.666656494	8	8	3.748733521	3.917922974	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-13	3	3	8.433334351	8	8	3.748733521	4.68460083	0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-14	3	3	7	8	8	3.748733521	3.251266479	0.03	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-15	3	3	6.766662598	8	8	3.748733521	3.017929077	0.05	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-16	3	3	8.300003052	8	8	3.748733521	4.551269531	<0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-17	3	3	5.933334351	8	8	3.748733521	2.18460083	0.12	Do Not Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-18	3	3	7.266662598	8	8	3.748733521	3.517929077	0.01	Reject H0
SS	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-19	3	3	16	8	8	3.748733521	12.25126648	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-01	3	3	21.33332825	8	8	14.92500305	6.408325195	0.14	Do Not Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-10	3	3	23	8	8	14.92500305	8.074996948	0.09	Do Not Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-02	3	3	27	8	8	14.92500305	12.07499695	0.02	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-03	3	3	26.66665649	8	8	14.92500305	11.74165344	0.02	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-04	3	3	56	8	8	14.92500305	41.07499695	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-05	3	3	24.66667175	8	8	14.92500305	9.741668701	0.07	Do Not Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-06	3	3	36.66665649	8	8	14.92500305	21.74165344	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-07	3	3	49	8	8	14.92500305	34.07499695	0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-09	3	3	28.33334351	8	8	14.92500305	13.40834045	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-11	3	3	33.33332825	8	8	14.92500305	18.4083252	0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-12	3	3	48.66667175	8	8	14.92500305	33.7416687	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-13	3	3	360	8	8	14.92500305	345.0749969	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-14	3	3	24.66665649	8	8	14.92500305	9.741653442	0.06	Do Not Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-15	3	3	31.33334351	8	8	14.92500305	16.40834045	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-16	3	3	36.66665649	8	8	14.92500305	21.74165344	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-17	3	3	191	8	8	14.92500305	176.0749969	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-18	3	3	913.3333282	8	8	14.92500305	898.4083252	<0.01	Reject H0
SS	Copper	CU	7440-50-8	mg/kg	IINY-DU-19	3	3	150	8	8	14.92500305	135.0749969	0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-01	3	3	0.030334473	8	7	0.062042236	-0.031707764	0.76	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-02	3	3	0.316665649	8	7	0.062042236	0.254623413	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-03	3	3	0.076324463	8	7	0.062042236	0.014282227	0.34	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-04	3	3	0.279998779	8	7	0.062042236	0.217956543	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-05	3	3	0.054000854	8	7	0.062042236	-0.008041382	0.61	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-09	3	3	0.030990601	8	7	0.062042236	-0.031051636	0.77	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-11	3	3	0.189987183	8	7	0.062042236	0.127944946	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-12	3	3	0.316665649	8	7	0.062042236	0.254623413	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-13	3	3	1.966659546	8	7	0.062042236	1.90461731	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-16	3	3	0.075683594	8	7	0.062042236	0.013641357	0.39	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-17	3	3	0.120986938	8	7	0.062042236	0.058944702	0.08	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-18	3	3	0.153335571	8	7	0.062042236	0.091293335	0.03	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-19	3	3	0.313323975	8	7	0.062042236	0.251281738	<0.01	Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-20	3	3	0.028671265	8	7	0.062042236	-0.033370972	0.78	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-21	3	3	0.024337769	8	7	0.062042236	-0.037704468	0.86	Do Not Reject H0
SS	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-22	3	3	0.270004272	8	7	0.062042236	0.207962036	<0.01	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-01	3	3	0.233337402	8	8	0.907501221	-0.674163818	0.91	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-02	3	3	3.633346558	8	8	0.907501221	2.725845337	<0.01	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-03	3	3	0.726669312	8	8	0.907501221	-0.180831909	0.61	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-04	3	3	4.466674805	8	8	0.907501221	3.559173584	0.03	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-05	3	3	0.539993286	8	8	0.907501221	-0.367507935	0.73	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-09	3	3	0.350006104	8	8	0.907501221	-0.557495117	0.85	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-11	3	3	1.03666687	8	8	0.907501221	0.129165649	0.41	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-12	3	3	3.866668701	8	8	0.907501221	2.95916748	<0.01	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-13	3	3	31	8	8	0.907501221	30.09249878	<0.01	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-16	3	3	0.790008545	8	8	0.907501221	-0.117492676	0.59	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-17	3	3	1.213348389	8	8	0.907501221	0.305847168	0.28	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-18	3	3	1.290008545	8	8	0.907501221	0.382507324	0.24	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-19	3	3	2.433334351	8	8	0.907501221	1.52583313	0.05	Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-20	3	3	0.290008545	8	8	0.907501221	-0.617492676	0.89	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-21	3	3	0.269989014	8	8	0.907501221	-0.637512207	0.92	Do Not Reject H0
SS	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-22	3	3	2.599990845	8	8	0.907501221	1.692489624	0.02	Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-01	3	3	0.008239746	8	8	0.055587769	-0.047348022	0.97	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-02	3	3	0.150009155	8	8	0.055587769	0.094421387	0.01	Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-03	3	3	0.027999878	8	8	0.055587769	-0.027587891	0.92	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-04	3	3	0.140991211	8	8	0.055587769	0.085403442	0.08	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-05	3	3	0.018325806	8	8	0.055587769	-0.037261963	0.96	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-09	3	3	0.013290405	8	8	0.055587769	-0.042297363	0.95	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-11	3	3	0.043991089	8	8	0.055587769	-0.01159668	0.67	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-12	3	3	0.346664429	8	8	0.055587769	0.29107666	<0.01	Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-13	3	3	3.033340454	8	8	0.055587769	2.977752686	<0.01	Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-16	3	3	0.075668335	8	8	0.055587769	0.020080566	0.18	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-17	3	3	0.04598999	8	8	0.055587769	-0.009597778	0.67	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-18	3	3	0.066665649	8	8	0.055587769	0.011077881	0.29	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-19	3	3	0.04800415	8	8	0.055587769	-0.007583618	0.66	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-20	3	3	0.015258789	8	8	0.055587769	-0.040328979	0.95	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-21	3	3	0.014831543	8	8	0.055587769	-0.040756226	0.98	Do Not Reject H0
SS	Fluorene	FL	86-73-7	mg/kg	IINY-DU-22	3	3	0.106338501	8	8	0.055587769	0.050750732	0.02	Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-01	3	3	0.136657715	8	8	0.287521362	-0.150863647	0.77	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-02	3	3	1.14666748	8	8	0.287521362	0.859146118	<0.01	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-03	3	3	0.306671143	8	8	0.287521362	0.01914978	0.45	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-04	3	3	1.033340454	8	8	0.287521362	0.745819092	<0.01	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-05	3	3	0.210006714	8	8	0.287521362	-0.077514648	0.64	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-09	3	3	0.14666748	8	8	0.287521362	-0.140853882	0.72	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-11	3	3	0.873336792	8	8	0.287521362	0.58581543	0.02	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-12	3	3	1.220001221	8	8	0.287521362	0.932479858	<0.01	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-13	3	3	7.333328247	8	8	0.287521362	7.045806885	<0.01	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-16	3	3	0.306671143	8	8	0.287521362	0.01914978	0.43	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-17	3	3	0.399993896	8	8	0.287521362	0.112472534	0.27	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-18	3	3	0.490005493	8	8	0.287521362	0.202484131	0.15	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-19	3	3	1.030014038	8	8	0.287521362	0.742492676	<0.01	Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-20	3	3	0.133331299	8	8	0.287521362	-0.154190063	0.77	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-21	3	3	0.098007202	8	8	0.287521362	-0.18951416	0.88	Do Not Reject H0
SS	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-22	3	3	0.949996948	8	8	0.287521362	0.662475586	0.02	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-01	3	3	15666.66667	8	8	14200	1466.666672	0.29	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-02	3	3	18666.66667	8	8	14200	4466.666672	0.03	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-03	3	3	18666.66667	8	8	14200	4466.666672	0.05	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-04	3	3	19333.33333	8	8	14200	5133.333328	0.04	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-05	3	3	19666.66667	8	8	14200	5466.666672	0.03	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-06	3	3	17333.33334	8	8	14200	3133.333344	0.12	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-07	3	3	18666.66667	8	8	14200	4466.666672	0.04	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-09	3	3	16333.33333	8	8	14200	2133.333328	0.19	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-10	3	3	17333.33333	8	8	14200	3133.333328	0.11	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-11	3	3	18333.33333	8	8	14200	4133.333328	0.05	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-12	3	3	17000	8	8	14200	2800	0.15	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-13	3	3	18333.33333	8	8	14200	4133.333328	0.04	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-14	3	3	16333.33333	8	8	14200	2133.333328	0.21	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-15	3	3	18333.33333	8	8	14200	4133.333328	0.03	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-16	3	3	21000	8	8	14200	6800	0.03	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-17	3	3	16333.33333	8	8	14200	2133.333328	0.23	Do Not Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-18	3	3	32333.33333	8	8	14200	18133.33333	<0.01	Reject H0
SS	Iron	FE	7439-89-6	mg/kg	IINY-DU-19	3	3	29333.33333	8	8	14200	15133.33333	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-01	3	3	64.33332825	8	8	55	9.333328247	0.27	Do Not Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-02	3	3	93.66667175	8	8	55	38.66667175	0.02	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-03	3	3	130	8	8	55	75	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-04	3	3	296.6666718	8	8	55	241.6666718	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-05	3	3	86	8	8	55	31	0.02	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-06	3	3	98.33332825	8	8	55	43.33332825	0.02	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-07	3	3	1086.666672	8	8	55	1031.666672	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-09	3	3	58.33334351	8	8	55	3.333343506	0.31	Do Not Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-10	3	3	34.33334351	8	8	55	-20.66665649	1.00	Do Not Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-11	3	3	123.3333282	8	8	55	68.33332825	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-12	3	3	84.66667175	8	8	55	29.66667175	0.04	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-13	3	3	166.6666718	8	8	55	111.6666718	0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-14	3	3	108.6666565	8	8	55	53.66665649	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-15	3	3	126.6666718	8	8	55	71.66667175	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-16	3	3	42	8	8	55	-13	0.94	Do Not Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-17	3	3	1866.666672	8	8	55	1811.666672	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-18	3	3	1433.333328	8	8	55	1378.333328	<0.01	Reject H0
SS	Lead	PB	7439-92-1	mg/kg	IINY-DU-19	3	3	55.33332825	8	8	55	0.333328247	0.41	Do Not Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-01	3	3	4266.666656	8	8	1712.5	2554.166656	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-10	3	3	10300	8	8	1712.5	8587.5	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-02	3	3	8766.666656	8	8	1712.5	7054.166656	0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-03	3	3	6066.666672	8	8	1712.5	4354.166672	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-04	3	3	6466.666656	8	8	1712.5	4754.166656	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-05	3	3	4966.666656	8	8	1712.5	3254.166656	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-06	3	3	10333.33333	8	8	1712.5	8620.833328	0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-07	3	3	11000	8	8	1712.5	9287.5	0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-09	3	3	6000	8	8	1712.5	4287.5	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-11	3	3	18000	8	8	1712.5	16287.5	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-12	3	3	7633.333344	8	8	1712.5	5920.833344	0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-13	3	3	5533.333344	8	8	1712.5	3820.833344	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-14	3	3	6466.666672	8	8	1712.5	4754.166672	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-15	3	3	6533.333344	8	8	1712.5	4820.833344	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-16	3	3	6266.666672	8	8	1712.5	4554.166672	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-17	3	3	35000	8	8	1712.5	33287.5	<0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-18	3	3	7866.666672	8	8	1712.5	6154.166672	0.01	Reject H0
SS	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-19	3	3	6900	8	8	1712.5	5187.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-01	3	3	473.3333282	8	8	211.5	261.8333282	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-02	3	3	640	8	8	211.5	428.5	0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-03	3	3	626.6666565	8	8	211.5	415.1666565	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-04	3	3	473.3333282	8	8	211.5	261.8333282	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-05	3	3	730	8	8	211.5	518.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-06	3	3	690	8	8	211.5	478.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-07	3	3	523.3333282	8	8	211.5	311.8333282	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-09	3	3	493.3333282	8	8	211.5	281.8333282	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-10	3	3	546.6666718	8	8	211.5	335.1666718	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-11	3	3	600	8	8	211.5	388.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-12	3	3	850	8	8	211.5	638.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-13	3	3	976.6666565	8	8	211.5	765.1666565	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-14	3	3	696.6666718	8	8	211.5	485.1666718	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-15	3	3	440	8	8	211.5	228.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-16	3	3	440	8	8	211.5	228.5	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-17	3	3	406.6666565	8	8	211.5	195.1666565	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-18	3	3	513.3333282	8	8	211.5	301.8333282	<0.01	Reject H0
SS	Manganese	MN	7439-96-5	mg/kg	IINY-DU-19	3	3	430	8	8	211.5	218.5	<0.01	Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-01	3	3	0.080657959	8	8	0.227493286	-0.146835327	1.00	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-02	3	3	0.09765625	8	8	0.227493286	-0.129837036	1.00	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-03	3	3	0.126663208	8	8	0.227493286	-0.100830078	0.98	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-04	3	3	0.286651611	8	8	0.227493286	0.059158325	0.13	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-05	3	3	0.153335571	8	8	0.227493286	-0.074157715	0.92	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-09	3	3	0.076004028	8	8	0.227493286	-0.151489258	1.00	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-11	3	3	0.183334351	8	8	0.227493286	-0.044158936	0.81	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-12	3	3	0.243331909	8	8	0.227493286	0.015838623	0.40	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-13	3	3	0.316665649	8	8	0.227493286	0.089172363	0.07	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-14	3	3	0.17666626	8	8	0.227493286	-0.050827026	0.82	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-15	3	3	0.21333313	8	8	0.227493286	-0.014160156	0.63	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-16	3	3	0.096328735	8	8	0.227493286	-0.131164551	1.00	Do Not Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-17	3	3	0.78666687	8	8	0.227493286	0.559173584	<0.01	Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-18	3	3	2.700012207	8	8	0.227493286	2.472518921	<0.01	Reject H0
SS	Mercury	HG	7439-97-6	mg/kg	IINY-DU-19	3	3	0.333343506	8	8	0.227493286	0.10585022	0.13	Do Not Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-01	3	3	0.008270264	8	8	0.017593384	-0.00932312	0.95	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-02	3	3	0.055664063	8	8	0.017593384	0.038070679	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-03	3	3	0.015991211	8	8	0.017593384	-0.001602173	0.57	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-04	3	1	0.050994873	8	8	0.017593384	0.033401489	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-05	3	3	0.01399231	8	8	0.017593384	-0.003601074	0.76	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-09	3	3	0.011245728	8	8	0.017593384	-0.006347656	0.94	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-11	3	3	0.085998535	8	8	0.017593384	0.068405151	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-12	3	3	0.151992798	8	8	0.017593384	0.134399414	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-13	3	3	0.933334351	8	8	0.017593384	0.915740967	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-16	3	3	0.054992676	8	8	0.017593384	0.037399292	0.05	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-17	3	3	0.078674316	8	8	0.017593384	0.061080933	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-18	3	3	0.226669312	8	8	0.017593384	0.209075928	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-19	3	3	0.065673828	8	8	0.017593384	0.048080444	<0.01	Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-20	3	3	0.013320923	8	8	0.017593384	-0.004272461	0.82	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-21	3	3	0.008987427	8	8	0.017593384	-0.008605957	0.97	Do Not Reject H0
SS	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-22	3	3	0.060653687	8	8	0.017593384	0.043060303	<0.01	Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-01	3	3	14	8	8	13.02500916	0.974990845	0.41	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-02	3	3	17	8	8	13.02500916	3.974990845	0.24	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-03	3	3	16.66667175	8	8	13.02500916	3.641662598	0.23	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-04	3	3	15.66665649	8	8	13.02500916	2.641647339	0.28	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-05	3	3	18	8	8	13.02500916	4.974990845	0.20	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-06	3	3	15	8	8	13.02500916	1.974990845	0.32	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-07	3	3	14	8	8	13.02500916	0.974990845	0.40	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-09	3	3	14.66667175	8	8	13.02500916	1.641662598	0.39	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-10	3	3	15.66667175	8	8	13.02500916	2.641662598	0.30	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-11	3	3	16	8	8	13.02500916	2.974990845	0.25	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-12	3	3	15	8	8	13.02500916	1.974990845	0.33	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-13	3	3	16.33332825	8	8	13.02500916	3.308319092	0.29	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-14	3	3	15.66667175	8	8	13.02500916	2.641662598	0.29	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-15	3	3	14.33332825	8	8	13.02500916	1.308319092	0.37	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-16	3	3	16	8	8	13.02500916	2.974990845	0.26	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-17	3	3	13.96665955	8	8	13.02500916	0.941650391	0.49	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-18	3	3	25	8	8	13.02500916	11.97499084	0.07	Do Not Reject H0
SS	Nickel	NI	7440-02-0	mg/kg	IINY-DU-19	3	3	16.66667175	8	8	13.02500916	3.641662598	0.27	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-01	3	3	0.082672119	8	8	0.43762207	-0.354949951	0.97	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-02	3	3	1.899993896	8	8	0.43762207	1.462371826	<0.01	Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-03	3	3	0.36000061	8	8	0.43762207	-0.07762146	0.61	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-04	3	3	1.953338623	8	8	0.43762207	1.515716553	0.02	Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-05	3	3	0.240005493	8	8	0.43762207	-0.197616577	0.75	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-09	3	3	0.125	8	8	0.43762207	-0.31262207	0.91	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-11	3	3	0.35333252	8	8	0.43762207	-0.084289551	0.60	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-12	3	3	2.733337402	8	8	0.43762207	2.295715332	<0.01	Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-13	3	3	22.66665649	8	8	0.43762207	22.22903442	<0.01	Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-16	3	3	0.533340454	8	8	0.43762207	0.095718384	0.34	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-17	3	3	0.543334961	8	8	0.43762207	0.105712891	0.32	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-18	3	3	0.766662598	8	8	0.43762207	0.329040527	0.09	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-19	3	3	0.866668701	8	8	0.43762207	0.429046631	0.12	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-20	3	3	0.123336792	8	8	0.43762207	-0.314285278	0.93	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-21	3	3	0.147994995	8	8	0.43762207	-0.289627075	0.90	Do Not Reject H0
SS	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-22	3	3	1.069992065	8	8	0.43762207	0.632369995	0.03	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-01	3	3	1200	8	8	727.5	472.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-02	3	3	1300	8	8	727.5	572.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-03	3	3	1300	8	8	727.5	572.5	0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-04	3	3	1266.666672	8	8	727.5	539.1666718	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-05	3	3	1233.333328	8	8	727.5	505.8333282	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-06	3	3	1200	8	8	727.5	472.5	0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-07	3	3	1033.333328	8	8	727.5	305.8333282	0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-09	3	3	1266.666672	8	8	727.5	539.1666718	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-10	3	3	1333.333328	8	8	727.5	605.8333282	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-11	3	3	956.6666565	8	8	727.5	229.1666565	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-12	3	3	1100	8	8	727.5	372.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-13	3	3	1400	8	8	727.5	672.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-14	3	3	993.3333282	8	8	727.5	265.8333282	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-15	3	3	1200	8	8	727.5	472.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-16	3	3	1566.666656	8	8	727.5	839.1666565	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-17	3	3	1200	8	8	727.5	472.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-18	3	3	1840	8	8	727.5	1112.5	<0.01	Reject H0
SS	Potassium	K	9/7/7440	mg/kg	IINY-DU-19	3	3	1433.333328	8	8	727.5	705.8333282	<0.01	Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-01	3	3	0.233337402	8	8	0.762481689	-0.529144287	0.88	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-02	3	3	3.566665649	8	8	0.762481689	2.80418396	<0.01	Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-03	3	3	0.730010986	8	8	0.762481689	-0.032470703	0.51	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-04	3	3	3.433334351	8	8	0.762481689	2.670852661	0.01	Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-05	3	3	0.576675415	8	8	0.762481689	-0.185806274	0.61	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-09	3	3	0.326675415	8	8	0.762481689	-0.435806274	0.84	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-11	3	3	1.223327637	8	8	0.762481689	0.460845947	0.15	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-12	3	3	3.200012207	8	8	0.762481689	2.437530518	<0.01	Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-13	3	3	24.66667175	8	8	0.762481689	23.90419006	<0.01	Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-16	3	3	0.670013428	8	8	0.762481689	-0.092468262	0.55	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-17	3	3	1.206680298	8	8	0.762481689	0.444198608	0.15	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-18	3	3	1.223327637	8	8	0.762481689	0.460845947	0.17	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-19	3	3	1.966674805	8	8	0.762481689	1.204193115	0.05	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-20	3	3	0.276672363	8	8	0.762481689	-0.485809326	0.86	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-21	3	3	0.283340454	8	8	0.762481689	-0.479141235	0.85	Do Not Reject H0
SS	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-22	3	3	2.633331299	8	8	0.762481689	1.870849609	0.01	Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-01	3	3	0.356658936	8	8	0.959991455	-0.60333252	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-02	3	3	0.319992065	8	8	0.959991455	-0.63999939	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-03	3	3	0.416671753	8	8	0.959991455	-0.543319702	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-04	3	3	0.486679077	8	8	0.959991455	-0.473312378	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-05	3	3	0.28666687	8	8	0.959991455	-0.673324585	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-06	3	3	0.350006104	8	8	0.959991455	-0.609985352	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-07	3	3	0.386672974	8	8	0.959991455	-0.573318481	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-09	3	3	0.289993286	8	8	0.959991455	-0.669998169	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-10	3	3	0.276657104	8	8	0.959991455	-0.683334351	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-11	3	3	0.550003052	8	8	0.959991455	-0.409988403	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-12	3	3	0.429992676	8	8	0.959991455	-0.529998779	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-13	3	3	0.61000061	8	8	0.959991455	-0.349990845	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-14	3	3	0.386672974	8	8	0.959991455	-0.573318481	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-15	3	3	0.53666687	8	8	0.959991455	-0.423324585	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-16	3	3	0.383331299	8	8	0.959991455	-0.576660156	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-17	3	3	0.466659546	8	8	0.959991455	-0.493331909	1.00	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-18	3	3	0.559997559	8	8	0.959991455	-0.399993896	0.98	Do Not Reject H0
SS	Selenium	SE	7782-49-2	mg/kg	IINY-DU-19	3	3	0.403335571	8	8	0.959991455	-0.556655884	1.00	Do Not Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-01	3	0	0.099	8	8	0.272491455	-0.173491455	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-02	3	0	0.1	8	8	0.272491455	-0.172491455	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-03	3	1	0.096008301	8	8	0.272491455	-0.176483154	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-04	3	3	0.102661133	8	8	0.272491455	-0.169830322	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-05	3	0	0.099	8	8	0.272491455	-0.173491455	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-06	3	3	0.07699585	8	8	0.272491455	-0.195495605	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-07	3	3	0.099990845	8	8	0.272491455	-0.17250061	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-09	3	0	0.095	8	8	0.272491455	-0.177491455	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-10	3	1	0.059005737	8	8	0.272491455	-0.213485718	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-11	3	3	0.079666138	8	8	0.272491455	-0.192825317	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-12	3	3	0.070999146	8	8	0.272491455	-0.20149231	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-13	3	3	0.089660645	8	8	0.272491455	-0.182830811	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-14	3	3	0.076675415	8	8	0.272491455	-0.19581604	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-15	3	3	0.080337524	8	8	0.272491455	-0.192153931	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-16	3	3	0.058334351	8	8	0.272491455	-0.214157104	1.00	Do Not Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-17	3	3	0.623336792	8	8	0.272491455	0.350845337	<0.01	Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-18	3	3	0.800003052	8	8	0.272491455	0.527511597	<0.01	Reject H0
SS	Silver	AG	7440-22-4	mg/kg	IINY-DU-19	3	3	0.123336792	8	8	0.272491455	-0.149154663	1.00	Do Not Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-01	3	3	101.6666565	8	8	43.75	57.91665649	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-02	3	3	73.66667175	8	8	43.75	29.91667175	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-03	3	3	88.66665649	8	8	43.75	44.91665649	0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-04	3	3	210	8	8	43.75	166.25	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-05	3	3	61.66667175	8	8	43.75	17.91667175	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-06	3	3	84.66667175	8	8	43.75	40.91667175	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-07	3	3	90.66667175	8	8	43.75	46.91667175	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-09	3	3	163.3333282	8	8	43.75	119.5833282	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-10	3	2	85.5	8	8	43.75	41.75	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-11	3	3	60.33332825	8	8	43.75	16.58332825	0.02	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-12	3	3	69.33334351	8	8	43.75	25.58334351	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-13	3	3	123.3333282	8	8	43.75	79.58332825	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-14	3	3	71.66665649	8	8	43.75	27.91665649	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-15	3	3	77.66665649	8	8	43.75	33.91665649	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-16	3	3	376.6666718	8	8	43.75	332.9166718	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-17	3	3	420	8	8	43.75	376.25	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-18	3	3	736.6666718	8	8	43.75	692.9166718	<0.01	Reject H0
SS	Sodium	NA	7440-23-5	mg/kg	IINY-DU-19	3	3	2300	8	8	43.75	2256.25	<0.01	Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-01	3	3	0.126663208	8	8	0.129241943	-0.002578735	0.52	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-02	3	3	0.130004883	8	8	0.129241943	0.000762939	0.50	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-03	3	3	0.133331299	8	8	0.129241943	0.004089355	0.45	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-04	3	3	0.183334351	8	8	0.129241943	0.054092407	0.06	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-05	3	3	0.10333252	8	8	0.129241943	-0.025909424	0.74	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-06	3	3	0.136657715	8	8	0.129241943	0.007415771	0.45	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-07	3	3	0.203338623	8	8	0.129241943	0.07409668	0.01	Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-09	3	3	0.123336792	8	8	0.129241943	-0.005905151	0.62	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-10	3	3	0.149993896	8	8	0.129241943	0.020751953	0.29	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-11	3	3	0.210006714	8	8	0.129241943	0.080764771	0.02	Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-12	3	3	0.14666748	8	8	0.129241943	0.017425537	0.30	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-13	3	3	0.166671753	8	8	0.129241943	0.03742981	0.17	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-14	3	3	0.120010376	8	8	0.129241943	-0.009231567	0.63	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-15	3	3	0.156661987	8	8	0.129241943	0.027420044	0.24	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-16	3	3	0.123336792	8	8	0.129241943	-0.005905151	0.57	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-17	3	3	0.123321533	8	8	0.129241943	-0.00592041	0.59	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-18	3	3	0.180007935	8	8	0.129241943	0.050765991	0.11	Do Not Reject H0
SS	Thallium	TL	7440-28-0	mg/kg	IINY-DU-19	3	3	0.126663208	8	8	0.129241943	-0.002578735	0.61	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-01	3	3	22	8	8	32.375	-10.375	0.88	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-02	3	3	21.66667175	8	8	32.375	-10.70832825	0.89	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-03	3	3	24	8	8	32.375	-8.375	0.77	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-04	3	3	22	8	8	32.375	-10.375	0.86	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-05	3	3	19.66667175	8	8	32.375	-12.70832825	0.92	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-06	3	3	20.33334351	8	8	32.375	-12.04165649	0.86	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-07	3	3	20.33332825	8	8	32.375	-12.04167175	0.91	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-09	3	3	25	8	8	32.375	-7.375	0.80	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-10	3	3	22.66665649	8	8	32.375	-9.708343506	0.85	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-11	3	3	23	8	8	32.375	-9.375	0.84	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-12	3	3	27	8	8	32.375	-5.375	0.69	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-13	3	3	30.33332825	8	8	32.375	-2.041671753	0.61	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-14	3	3	20	8	8	32.375	-12.375	0.90	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-15	3	3	25.33332825	8	8	32.375	-7.041671753	0.73	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-16	3	3	29.33332825	8	8	32.375	-3.041671753	0.68	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-17	3	3	19	8	8	32.375	-13.375	0.93	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-18	3	3	27.33332825	8	8	32.375	-5.041671753	0.71	Do Not Reject H0
SS	Vanadium	V	7440-62-2	mg/kg	IINY-DU-19	3	3	47.33332825	8	8	32.375	14.95832825	0.08	Do Not Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-01	3	3	78.33332825	8	8	42.375	35.95832825	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-10	3	3	58	8	8	42.375	15.625	0.12	Do Not Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-02	3	3	75.33332825	8	8	42.375	32.95832825	0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-03	3	3	109	8	8	42.375	66.625	0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-04	3	3	206.6666718	8	8	42.375	164.2916718	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-05	3	3	75.66667175	8	8	42.375	33.29167175	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-06	3	3	67.66667175	8	8	42.375	25.29167175	0.04	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-07	3	3	206.6666718	8	8	42.375	164.2916718	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-09	3	3	68.66665649	8	8	42.375	26.29165649	0.03	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-11	3	3	99	8	8	42.375	56.625	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-12	3	3	96	8	8	42.375	53.625	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-13	3	3	153.3333435	8	8	42.375	110.9583435	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-14	3	3	75.33332825	8	8	42.375	32.95832825	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-15	3	3	163.3333282	8	8	42.375	120.9583282	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-16	3	3	69	8	8	42.375	26.625	0.02	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-17	3	3	7033.333328	8	8	42.375	6990.958328	0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-18	3	3	1161	8	8	42.375	1118.625	<0.01	Reject H0
SS	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-19	3	3	106.6666565	8	8	42.375	64.29165649	<0.01	Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SB	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-17	3	3	0.098342896	8	8	0.013412476	0.08493042	<0.01	Reject H0
SB	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-18	3	3	0.088668823	8	8	0.013412476	0.075256348	<0.01	Reject H0
SB	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-19	3	3	0.000976563	8	8	0.013412476	-0.012435913	1.00	Do Not Reject H0
SB	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-20	3	3	0.004928589	8	8	0.013412476	-0.008483887	0.96	Do Not Reject H0
SB	1-Methylnaphthalene	MTNPH1	90-12-0	mg/kg	IINY-DU-21	3	3	0.005599976	8	8	0.013412476	-0.0078125	0.97	Do Not Reject H0
SB	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-17	3	3	0.13999939	8	8	0.012390137	0.127609253	<0.01	Reject H0
SB	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-18	3	3	0.105010986	8	8	0.012390137	0.09262085	<0.01	Reject H0
SB	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-19	3	3	0.00151062	8	8	0.012390137	-0.010879517	1.00	Do Not Reject H0
SB	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-20	3	3	0.007553101	8	8	0.012390137	-0.004837036	0.94	Do Not Reject H0
SB	2-Methylnaphthalene	MTNPH2	91-57-6	mg/kg	IINY-DU-21	3	3	0.00793457	8	8	0.012390137	-0.004455566	0.90	Do Not Reject H0
SB	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-17	3	3	0.070999146	8	8	0.038757324	0.032241821	0.06	Do Not Reject H0
SB	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-18	3	3	0.196655273	8	8	0.038757324	0.157897949	<0.01	Reject H0
SB	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-19	3	3	0.001586914	8	8	0.038757324	-0.03717041	1.00	Do Not Reject H0
SB	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-20	3	3	0.006027222	8	8	0.038757324	-0.032730103	0.99	Do Not Reject H0
SB	Acenaphthene	ACNP	83-32-9	mg/kg	IINY-DU-21	3	3	0.004837036	8	8	0.038757324	-0.033920288	0.99	Do Not Reject H0
SB	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-17	3	3	0.036010742	8	8	0.033493042	0.0025177	0.38	Do Not Reject H0
SB	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-18	3	3	0.056655884	8	8	0.033493042	0.023162842	0.10	Do Not Reject H0
SB	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-19	3	3	0.005630493	8	8	0.033493042	-0.027862549	1.00	Do Not Reject H0
SB	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-20	3	3	0.021331787	8	8	0.033493042	-0.012161255	0.84	Do Not Reject H0
SB	Acenaphthylene	ACNPY	208-96-8	mg/kg	IINY-DU-21	3	3	0.011260986	8	8	0.033493042	-0.022232056	0.97	Do Not Reject H0
SB	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-17	3	3	6033.333328	8	8	12162.5	-6129.166672	1.00	Do Not Reject H0
SB	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-18	3	3	5000	8	8	12162.5	-7162.5	1.00	Do Not Reject H0
SB	Aluminum	AL	7429-90-5	mg/kg	IINY-DU-19	3	3	22000	8	8	12162.5	9837.5	<0.01	Reject H0
SB	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-17	3	3	0.15032959	8	8	0.059188843	0.091140747	0.03	Reject H0
SB	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-18	3	3	0.169998169	8	8	0.059188843	0.110809326	0.03	Reject H0
SB	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-19	3	3	0.005432129	8	8	0.059188843	-0.053756714	1.00	Do Not Reject H0
SB	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-20	3	3	0.026000977	8	8	0.059188843	-0.033187866	0.89	Do Not Reject H0
SB	Anthracene	ANTH	120-12-7	mg/kg	IINY-DU-21	3	3	0.015670776	8	8	0.059188843	-0.043518066	0.96	Do Not Reject H0
SB	Antimony	SB	7440-36-0	mg/kg	IINY-DU-17	3	3	4.566665649	8	8	0.986236572	3.580429077	<0.01	Reject H0
SB	Antimony	SB	7440-36-0	mg/kg	IINY-DU-18	3	3	9.833343506	8	8	0.986236572	8.847106934	<0.01	Reject H0
SB	Antimony	SB	7440-36-0	mg/kg	IINY-DU-19	3	3	0.113327026	8	8	0.986236572	-0.872909546	1.00	Do Not Reject H0
SB	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-17	3	3	23.33334351	8	8	5.325012207	18.0083313	<0.01	Reject H0
SB	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-18	3	3	17.33334351	8	8	5.325012207	12.0083313	0.01	Reject H0
SB	Arsenic	AS	7440-38-2	mg/kg	IINY-DU-19	3	3	2.766662598	8	8	5.325012207	-2.558349609	0.97	Do Not Reject H0
SB	Barium	BA	7440-39-3	mg/kg	IINY-DU-17	3	3	356.6666565	8	8	33.375	323.2916565	<0.01	Reject H0
SB	Barium	BA	7440-39-3	mg/kg	IINY-DU-18	3	3	303.3333435	8	8	33.375	269.9583435	<0.01	Reject H0
SB	Barium	BA	7440-39-3	mg/kg	IINY-DU-19	3	3	35.33334351	8	8	33.375	1.958343506	0.44	Do Not Reject H0
SB	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-17	3	3	0.456665039	8	8	0.307479858	0.149185181	0.20	Do Not Reject H0
SB	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-18	3	3	0.503341675	8	8	0.307479858	0.195861816	0.15	Do Not Reject H0
SB	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-19	3	3	0.022003174	8	8	0.307479858	-0.285476685	1.00	Do Not Reject H0
SB	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-20	3	3	0.072998047	8	8	0.307479858	-0.234481812	0.91	Do Not Reject H0
SB	Benzo(a)anthracene	BZAA	56-55-3	mg/kg	IINY-DU-21	3	3	0.066665649	8	8	0.307479858	-0.240814209	0.95	Do Not Reject H0
SB	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-17	3	3	0.289993286	8	8	0.329742432	-0.039749146	0.58	Do Not Reject H0
SB	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-18	3	3	0.356658936	8	8	0.329742432	0.026916504	0.43	Do Not Reject H0
SB	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-19	3	3	0.025009155	8	8	0.329742432	-0.304733276	1.00	Do Not Reject H0
SB	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-20	3	3	0.074661255	8	8	0.329742432	-0.255081177	0.97	Do Not Reject H0
SB	Benzo(a)pyrene	BZAP	50-32-8	mg/kg	IINY-DU-21	3	3	0.067657471	8	8	0.329742432	-0.262084961	0.95	Do Not Reject H0
SB	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-17	3	3	0.653335571	8	8	0.523986816	0.129348755	0.35	Do Not Reject H0
SB	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-18	3	3	0.836669922	8	8	0.523986816	0.312683105	0.17	Do Not Reject H0
SB	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-19	3	3	0.038330078	8	8	0.523986816	-0.485656738	1.00	Do Not Reject H0
SB	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-20	3	3	0.113327026	8	8	0.523986816	-0.41065979	0.97	Do Not Reject H0
SB	Benzo(b)fluoranthene	BZBF	205-99-2	mg/kg	IINY-DU-21	3	3	0.123336792	8	8	0.523986816	-0.400650024	0.97	Do Not Reject H0
SB	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-17	3	3	0.246673584	8	8	0.261871338	-0.015197754	0.54	Do Not Reject H0
SB	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-18	3	3	0.296661377	8	8	0.261871338	0.034790039	0.39	Do Not Reject H0
SB	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-19	3	3	0.022674561	8	8	0.261871338	-0.239196777	1.00	Do Not Reject H0
SB	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-20	3	3	0.045669556	8	8	0.261871338	-0.216201782	0.96	Do Not Reject H0
SB	Benzo(g,h,i)perylene	BZGHIP	191-24-2	mg/kg	IINY-DU-21	3	3	0.040664673	8	8	0.261871338	-0.221206665	0.98	Do Not Reject H0
SB	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-17	3	3	0.15032959	8	8	0.191375732	-0.041046143	0.63	Do Not Reject H0
SB	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-18	3	3	0.164993286	8	8	0.191375732	-0.026382446	0.55	Do Not Reject H0
SB	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-19	3	3	0.011734009	8	8	0.191375732	-0.179641724	1.00	Do Not Reject H0
SB	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-20	3	3	0.033660889	8	8	0.191375732	-0.157714844	0.96	Do Not Reject H0
SB	Benzo(k)fluoranthene	BZKF	207-08-9	mg/kg	IINY-DU-21	3	3	0.038330078	8	8	0.191375732	-0.153045654	0.95	Do Not Reject H0
SB	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-17	3	3	1.333328247	8	8	0.43963623	0.893692017	<0.01	Reject H0
SB	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-18	3	3	0.653335571	8	8	0.43963623	0.213699341	0.13	Do Not Reject H0
SB	Beryllium	BE	7440-41-7	mg/kg	IINY-DU-19	3	3	0.183334351	8	8	0.43963623	-0.25630188	0.91	Do Not Reject H0
SB	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-17	3	3	3.5	8	8	0.174987793	3.325012207	<0.01	Reject H0
SB	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-18	3	3	12.09999084	8	8	0.174987793	11.92500305	<0.01	Reject H0
SB	Cadmium	CD	7440-43-9	mg/kg	IINY-DU-19	3	3	0.092330933	8	8	0.174987793	-0.08265686	1.00	Do Not Reject H0
SB	Calcium	CA	7440-70-2	mg/kg	IINY-DU-17	3	3	18333.33333	8	8	1222.5	17110.83333	<0.01	Reject H0
SB	Calcium	CA	7440-70-2	mg/kg	IINY-DU-18	3	3	32000	8	8	1222.5	30777.5	<0.01	Reject H0
SB	Calcium	CA	7440-70-2	mg/kg	IINY-DU-19	3	3	18000	8	8	1222.5	16777.5	<0.01	Reject H0
SB	Chromium	CR	7440-47-3	mg/kg	IINY-DU-17	3	3	12.66667175	8	8	14.6625061	-1.995834351	0.66	Do Not Reject H0
SB	Chromium	CR	7440-47-3	mg/kg	IINY-DU-18	3	3	31.33332825	8	8	14.6625061	16.67082214	<0.01	Reject H0
SB	Chromium	CR	7440-47-3	mg/kg	IINY-DU-19	3	3	7.766662598	8	8	14.6625061	-6.895843506	0.77	Do Not Reject H0
SB	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-17	3	3	0.759994507	8	8	0.424224854	0.335769653	0.09	Do Not Reject H0
SB	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-18	3	3	1.149993896	8	8	0.424224854	0.725769043	0.03	Reject H0
SB	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-19	3	3	0.024658203	8	8	0.424224854	-0.39956665	1.00	Do Not Reject H0
SB	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-20	3	3	0.079330444	8	8	0.424224854	-0.344894409	0.97	Do Not Reject H0
SB	Chrysene	CHRYSENE	218-01-9	mg/kg	IINY-DU-21	3	3	0.078674316	8	8	0.424224854	-0.345550537	0.97	Do Not Reject H0
SB	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-17	3	3	6.566665649	8	8	3.748733521	2.817932129	0.06	Do Not Reject H0
SB	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-18	3	3	6.333328247	8	8	3.748733521	2.584594727	0.08	Do Not Reject H0
SB	Cobalt	CO	7440-48-4	mg/kg	IINY-DU-19	3	3	17	8	8	3.748733521	13.25126648	<0.01	Reject H0
SB	Copper	CU	7440-50-8	mg/kg	IINY-DU-17	3	3	146.6666718	8	8	14.92500305	131.7416687	<0.01	Reject H0
SB	Copper	CU	7440-50-8	mg/kg	IINY-DU-18	3	3	5400	8	8	14.92500305	5385.074997	0.01	Reject H0
SB	Copper	CU	7440-50-8	mg/kg	IINY-DU-19	3	3	210	8	8	14.92500305	195.0749969	<0.01	Reject H0
SB	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-17	3	3	0.115997314	8	7	0.062042236	0.053955078	0.07	Do Not Reject H0
SB	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-18	3	3	0.121322632	8	7	0.062042236	0.059280396	0.11	Do Not Reject H0
SB	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-19	3	3	0.005569458	8	7	0.062042236	-0.056472778	0.98	Do Not Reject H0
SB	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-20	3	3	0.013671875	8	7	0.062042236	-0.048370361	0.98	Do Not Reject H0
SB	Dibenz(a,h)anthracene	DBAHA	53-70-3	mg/kg	IINY-DU-21	3	3	0.011993408	8	7	0.062042236	-0.050048828	0.96	Do Not Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

Table 10 Background Comparison Permutation Tests

Matrix	Analyte Name	Analyte Code	CAS	Units	Site Data				Background Data			Permutation Test (H0: Site <= Background)	Right-Taile Prob.	Kaplan-Meier Conclusion
					DU	N	No. Detects	Mean [1]	N	No. Detects	Mean [1]	Site Mean - Background Mean		
SB	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-17	3	3	0.980010986	8	8	0.907501221	0.072509766	0.45	Do Not Reject H0
SB	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-18	3	3	1.016662598	8	8	0.907501221	0.109161377	0.37	Do Not Reject H0
SB	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-19	3	3	0.040664673	8	8	0.907501221	-0.866836548	1.00	Do Not Reject H0
SB	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-20	3	3	0.136672974	8	8	0.907501221	-0.770828247	0.98	Do Not Reject H0
SB	Fluoranthene	FLA	206-44-0	mg/kg	IINY-DU-21	3	3	0.126663208	8	8	0.907501221	-0.780838013	1.00	Do Not Reject H0
SB	Fluorene	FL	86-73-7	mg/kg	IINY-DU-17	3	3	0.083343506	8	8	0.055587769	0.027755737	0.11	Do Not Reject H0
SB	Fluorene	FL	86-73-7	mg/kg	IINY-DU-18	3	3	0.150009155	8	8	0.055587769	0.094421387	0.01	Reject H0
SB	Fluorene	FL	86-73-7	mg/kg	IINY-DU-19	3	3	0.002532959	8	8	0.055587769	-0.05305481	1.00	Do Not Reject H0
SB	Fluorene	FL	86-73-7	mg/kg	IINY-DU-20	3	3	0.008956909	8	8	0.055587769	-0.046630859	0.96	Do Not Reject H0
SB	Fluorene	FL	86-73-7	mg/kg	IINY-DU-21	3	3	0.005691528	8	8	0.055587769	-0.04989624	0.99	Do Not Reject H0
SB	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-17	3	3	0.208343506	8	8	0.287521362	-0.079177856	0.67	Do Not Reject H0
SB	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-18	3	3	0.256668091	8	8	0.287521362	-0.030853271	0.56	Do Not Reject H0
SB	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-19	3	3	0.023330688	8	8	0.287521362	-0.264190674	1.00	Do Not Reject H0
SB	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-20	3	3	0.048995972	8	8	0.287521362	-0.238525391	0.97	Do Not Reject H0
SB	Indeno(1,2,3-c,d)pyrene	INP123	193-39-5	mg/kg	IINY-DU-21	3	3	0.047332764	8	8	0.287521362	-0.240188599	0.95	Do Not Reject H0
SB	Iron	FE	7439-89-6	mg/kg	IINY-DU-17	3	3	25666.66667	8	8	14200	11466.66667	<0.01	Reject H0
SB	Iron	FE	7439-89-6	mg/kg	IINY-DU-18	3	3	37333.33333	8	8	14200	23133.33333	0.01	Reject H0
SB	Iron	FE	7439-89-6	mg/kg	IINY-DU-19	3	3	34666.66666	8	8	14200	20466.66666	0.01	Reject H0
SB	Lead	PB	7439-92-1	mg/kg	IINY-DU-17	3	3	1573.333328	8	8	55	1518.333328	<0.01	Reject H0
SB	Lead	PB	7439-92-1	mg/kg	IINY-DU-18	3	3	2433.333344	8	8	55	2378.333344	<0.01	Reject H0
SB	Lead	PB	7439-92-1	mg/kg	IINY-DU-19	3	3	12.33334351	8	8	55	-42.66665649	1.00	Do Not Reject H0
SB	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-17	3	3	8233.333328	8	8	1712.5	6520.833328	0.01	Reject H0
SB	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-18	3	3	17333.33333	8	8	1712.5	15620.83333	<0.01	Reject H0
SB	Magnesium	MG	7439-95-4	mg/kg	IINY-DU-19	3	3	7266.666656	8	8	1712.5	5554.166656	<0.01	Reject H0
SB	Manganese	MN	7439-96-5	mg/kg	IINY-DU-17	3	3	170	8	8	211.5	-41.5	0.74	Do Not Reject H0
SB	Manganese	MN	7439-96-5	mg/kg	IINY-DU-18	3	3	480	8	8	211.5	268.5	0.02	Reject H0
SB	Manganese	MN	7439-96-5	mg/kg	IINY-DU-19	3	3	276.6666718	8	8	211.5	65.16667175	0.15	Do Not Reject H0
SB	Mercury	HG	7439-97-6	mg/kg	IINY-DU-17	3	3	0.82333374	8	8	0.227493286	0.595840454	<0.01	Reject H0
SB	Mercury	HG	7439-97-6	mg/kg	IINY-DU-18	3	3	1.173339844	8	8	0.227493286	0.945846558	<0.01	Reject H0
SB	Mercury	HG	7439-97-6	mg/kg	IINY-DU-19	3	3	0.206665039	8	8	0.227493286	-0.020828247	0.63	Do Not Reject H0
SB	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-17	3	3	0.240005493	8	8	0.017593384	0.222412109	<0.01	Reject H0
SB	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-18	3	3	0.559997559	8	8	0.017593384	0.542404175	<0.01	Reject H0
SB	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-19	3	2	0.001998901	8	8	0.017593384	-0.015594482	1.00	Do Not Reject H0
SB	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-20	3	3	0.007507324	8	8	0.017593384	-0.01008606	0.96	Do Not Reject H0
SB	Naphthalene	NAPH	91-20-3	mg/kg	IINY-DU-21	3	3	0.007827759	8	8	0.017593384	-0.009765625	0.94	Do Not Reject H0
SB	Nickel	NI	7440-02-0	mg/kg	IINY-DU-17	3	3	19.66667175	8	8	13.02500916	6.641662598	0.23	Do Not Reject H0
SB	Nickel	NI	7440-02-0	mg/kg	IINY-DU-18	3	3	65	8	8	13.02500916	51.97499084	<0.01	Reject H0
SB	Nickel	NI	7440-02-0	mg/kg	IINY-DU-19	3	3	14	8	8	13.02500916	0.974990845	0.40	Do Not Reject H0
SB	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-17	3	3	0.820007324	8	8	0.43762207	0.382385254	0.09	Do Not Reject H0
SB	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-18	3	3	0.973327637	8	8	0.43762207	0.535705566	0.04	Reject H0
SB	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-19	3	3	0.018035889	8	8	0.43762207	-0.419586182	1.00	Do Not Reject H0
SB	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-20	3	3	0.069656372	8	8	0.43762207	-0.367965698	0.99	Do Not Reject H0
SB	Phenanthrene	PHAN	85-01-8	mg/kg	IINY-DU-21	3	3	0.056655884	8	8	0.43762207	-0.380966187	1.00	Do Not Reject H0
SB	Potassium	K	2023695	mg/kg	IINY-DU-17	3	3	573.3333435	8	8	727.5	-154.1666565	1.00	Do Not Reject H0
SB	Potassium	K	2023695	mg/kg	IINY-DU-18	3	3	723.3333282	8	8	727.5	-4.166671753	0.47	Do Not Reject H0
SB	Potassium	K	2023695	mg/kg	IINY-DU-19	3	3	1133.333328	8	8	727.5	405.8333282	<0.01	Reject H0
SB	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-17	3	3	0.82333374	8	8	0.762481689	0.060852051	0.44	Do Not Reject H0
SB	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-18	3	3	0.936676025	8	8	0.762481689	0.174194336	0.31	Do Not Reject H0
SB	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-19	3	3	0.039672852	8	8	0.762481689	-0.722808838	1.00	Do Not Reject H0
SB	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-20	3	3	0.13999939	8	8	0.762481689	-0.6224823	0.96	Do Not Reject H0
SB	Pyrene	PYR	129-00-0	mg/kg	IINY-DU-21	3	3	0.126663208	8	8	0.762481689	-0.635818481	0.99	Do Not Reject H0
SB	Selenium	SE	7782-49-2	mg/kg	IINY-DU-17	3	3	1.333343506	8	8	0.959991455	0.373352051	0.02	Reject H0
SB	Selenium	SE	7782-49-2	mg/kg	IINY-DU-18	3	3	0.866668701	8	8	0.959991455	-0.093322754	0.80	Do Not Reject H0
SB	Selenium	SE	7782-49-2	mg/kg	IINY-DU-19	3	3	0.229995728	8	8	0.959991455	-0.729995728	1.00	Do Not Reject H0
SB	Silver	AG	7440-22-4	mg/kg	IINY-DU-17	3	3	0.263336182	8	8	0.272491455	-0.009155273	0.56	Do Not Reject H0
SB	Silver	AG	7440-22-4	mg/kg	IINY-DU-18	3	3	1.476669312	8	8	0.272491455	1.204177856	<0.01	Reject H0
SB	Silver	AG	7440-22-4	mg/kg	IINY-DU-19	3	3	0.102325439	8	8	0.272491455	-0.170166016	1.00	Do Not Reject H0
SB	Sodium	NA	7440-23-5	mg/kg	IINY-DU-17	3	3	500	8	8	43.75	456.25	<0.01	Reject H0
SB	Sodium	NA	7440-23-5	mg/kg	IINY-DU-18	3	3	426.6666718	8	8	43.75	382.9166718	<0.01	Reject H0
SB	Sodium	NA	7440-23-5	mg/kg	IINY-DU-19	3	3	2733.333328	8	8	43.75	2689.583328	<0.01	Reject H0
SB	Thallium	TL	7440-28-0	mg/kg	IINY-DU-17	3	3	0.706680298	8	8	0.129241943	0.577438354	<0.01	Reject H0
SB	Thallium	TL	7440-28-0	mg/kg	IINY-DU-18	3	3	0.309997559	8	8	0.129241943	0.180755615	<0.01	Reject H0
SB	Thallium	TL	7440-28-0	mg/kg	IINY-DU-19	3	3	0.07232666	8	8	0.129241943	-0.056915283	0.93	Do Not Reject H0
SB	Vanadium	V	7440-62-2	mg/kg	IINY-DU-17	3	3	22.33334351	8	8	32.375	-10.04165649	0.84	Do Not Reject H0
SB	Vanadium	V	7440-62-2	mg/kg	IINY-DU-18	3	3	17.66667175	8	8	32.375	-14.70832825	0.94	Do Not Reject H0
SB	Vanadium	V	7440-62-2	mg/kg	IINY-DU-19	3	3	55.33332825	8	8	32.375	22.95832825	0.02	Reject H0
SB	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-17	3	3	2133.333328	8	8	42.375	2090.958328	<0.01	Reject H0
SB	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-18	3	3	4566.666672	8	8	42.375	4524.291672	<0.01	Reject H0
SB	Zinc	ZN	7440-66-6	mg/kg	IINY-DU-19	3	3	61	8	8	42.375	18.625	0.05	Reject H0

Note:

Permutation Test of Null Hypothesis H0: Site Mean <= Background Mean at 95% Significance Level

1. Kaplan-Meier estimate used for data sets with nondetect sample results.

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Table 12 Constituents Exceeding Levels at Each Decision Unit (Based on 95% UCLs) and Background Screening Criteria

Decision Unit	Soil Type	Constituent Exceeding Screening Criteria and Background Concentrations[1,2]		
		Ecological Soil Screening Level	EPA Residential Regional Screening Level	EPA Industrial Regional Screening Level
DU-1	Surface Soil	Arsenic, Cadmium, Manganese, Zinc	Arsenic, Cobalt, Manganese	Arsenic
DU-2	Surface Soil	Arsenic, Lead, Manganese, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene , Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Arsenic, Cobalt, Iron, Manganese, Thallium, Benzo(a)pyrene, Benzo(a)anthracene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Arsenic, Benzo(a)pyrene
DU-3	Surface Soil	Arsenic, Copper, Lead, Manganese, Zinc	Arsenic, Cobalt, Iron, Manganese	Arsenic
DU-4	Surface Soil	Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Zinc, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Arsenic, Cobalt, Iron, Lead, Manganese, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Arsenic
DU-5	Surface Soil	Arsenic, Lead, Manganese, Zinc	Arsenic, Cobalt, Iron, Manganese	Arsenic
DU-6	Surface Soil	Arsenic, Copper, Lead, Manganese	Arsenic, Cobalt, Manganese	Arsenic
DU-7	Surface Soil	Arsenic, Barium, Cadmium, Copper, Lead, Manganese, Thallium, Zinc	Arsenic, Iron, Lead, Manganese, Thallium	Arsenic, Lead
DU-9	Surface Soil	Copper , Manganese, Zinc	Cobalt, Manganese	No Exceedances
DU-10	Surface Soil	Manganese	Cobalt, Manganese	No Exceedances
DU-11	Surface Soil	Arsenic, Cadmium, Copper, Lead, Manganese, Thallium, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Chrysene, Indeno(1,2,3-c,d)pyrene, Total HMW PAHs	Arsenic, Cobalt, Manganese, Thallium, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic
DU-12	Surface Soil	Arsenic, Cadmium, Copper, Lead, Manganese, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene , Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Arsenic, Cobalt, Manganese, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)pyrene
DU-13	Surface Soil	Antimony, Arsenic, Cadmium, Copper, Lead, Manganese, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, dibenz(a,h)anthracene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total LMW PAHs, and Total HMW PAHs	Antimony, Arsenic, Cobalt, Copper, Iron, Manganese, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene
DU-14	Surface Soil	Arsenic, Lead, Manganese, Zinc	Arsenic, Cobalt, Manganese	Arsenic
DU-15	Surface Soil	Cadmium, Copper, Lead, Manganese, Zinc	Cobalt, Iron, Manganese	No Exceedances
DU-16	Surface Soil	Cadmium, Copper, Manganese, Zinc	Cobalt, Iron, Manganese	No Exceedances
DU-17	Surface Soil	Mercury, Antimony, Barium, Cadmium, Copper, Lead, Manganese, Zinc, Total PCBs, PCB-1248 (Aroclor 1248) , PCB-1260 (Aroclor 1260)	Mercury, Antimony, Cadmium, Copper, Lead, Manganese, Zinc	Lead
	Surface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Copper, Lead, Selenium, Thallium, Zinc	Antimony, Arsenic, Iron, Lead, Thallium, Zinc	Arsenic, Lead
DU-18	Surface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Total Chromium, Copper, Lead, Manganese, Zinc, Benzo(a)pyrene, Benzo(b)fluoranthene, Chrysene, Total PCBs, PCB-1248 (Aroclor 1248) , PCB-1260 (Aroclor 1260)	Mercury, Antimony, Arsenic, Cobalt, Copper, Iron, Lead, Manganese, Zinc, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Total PCBs, PCB-1260 (Aroclor 1260)	Mercury, Arsenic, Lead
	Subsurface Soil	Mercury, Antimony, Arsenic, Barium, Cadmium, Total Chromium, Copper, Lead, Manganese, Nickel, Thallium, Zinc, Chrysene, Total PCBs, PCB-1262 (Aroclor 1262)	Mercury, Antimony, Arsenic, Cadmium, Copper, Iron, Lead, Manganese, Thallium, Zinc	Copper, Arsenic, Lead
DU-19	Surface Soil	Arsenic, Cadmium, Cobalt, Copper, Manganese, Zinc, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Total HMW PAHs	Aluminum, Arsenic, Cobalt, Iron, Manganese, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene,	Arsenic, Benzo(a)pyrene
	Subsurface Soil	Cobalt, Copper, Vanadium, Zinc, Total PCBs, PCB-1260 (Aroclor 1260)	Aluminum, Cobalt, Iron, Vanadium	No Exceedances
DU-20	Surface Soil	No Exceedances	No Exceedances	No Exceedances
	Subsurface Soil	No Exceedances	No Exceedances	No Exceedances

Table 12 Constituents Exceeding Levels at Each Decision Unit (Based on 95% UCLs) and Background Screening Criteria

Decision Unit	Soil Type	Constituent Exceeding Screening Criteria and Background Concentrations[1,2]		
		Ecological Soil Screening Level	EPA Residential Regional Screening Level	EPA Industrial Regional Screening Level
DU-21	Surface Soil	No Exceedances	No Exceedances	No Exceedances
	Subsurface Soil	No Exceedances	No Exceedances	No Exceedances
DU-22	Surface Soil	Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene , Chrysene, Fluoranthene, Indeno(1,2,3-c,d)pyrene, Pyrene, Total HMW PAHs	Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene	Benzo(a)pyrene
DU-23	Surface Soil	Total PCBs, PCB-1260 (Aroclor 1260)	No Exceedances	No Exceedances
DU-25	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-1	Surface Soil	1,3-Dinitrobenzene (DNB)	No Exceedances	No Exceedances
EXDU-2	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-3	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-4	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-5	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-6	Surface Soil	No Exceedances	No Exceedances	No Exceedances
EXDU-7	Surface Soil	No Exceedances	No Exceedances	No Exceedances

1. United States Environmental Protection Agency (EPA) Regional Screening Levels (RSLs) Table, EPA, May 2020.
2. EPA Ecological Soil Screening Levels (ECO-SSLs), February 2005 - April 2008.

Note:
DU = Decision Unit
HMW = High molecular weight
LMW = Low molecular weight
PAH = Polycyclic aromatic hydrocarbon
PCB = Polychlorinated biphenyl

Table 13 Phase I Sediment Grain Size and Total Organic Carbon Analytical Results

	Sample Number	IINY-SD-01	IINY-SD-02	IINY-SD-03	IINY-SD-04	IINY-SD-05	IINY-SD-06	IINY-SD-07
	Sample ID	IINY-SD01-20200407	IINY-SD02-20200407	IINY-SD03-20200407	IINY-SD04-20200407	IINY-SD05-20200407	IINY-SD06-20200407	IINY-SD07-20200407
	Depth (in)	0-6	0-6	0-6	0-6	0-6	0-6	0-6
	Matrix	Sediment	Sediment	Sediment	Sediment	Sediment	Sediment	Sediment
	Location	Intertidal Marsh Shoreline	Intertidal Marsh Shoreline, Groundwater Seep	Intertidal Marsh Shoreline, Tidal Wetland Swale Outlet	Hudson River Shoreline, Macroinvertebrate Observation Area	Hudson River Shoreline	Hudson River Shoreline	Intertidal Marsh Shoreline, Drainage Swale Outlet
	Sample Date / Time	4/7/2020 10:40	4/7/2020 17:00	4/7/2020 16:00	4/7/2020 2:35	4/7/2020 3:19	4/7/2020 2:47	4/7/2020 16:30
Analyte	**Unit**							
Grain Size								
Gravel	Percent	0.00	8.76	0.00	22.73	2.89	4.28	0.00
Sand Coarse	Percent	0.00	1.82	0.00	14.84	1.08	14.46	0.00
Sand Medium	Percent	7.83	4.01	3.22	36.45	15.34	69.17	3.94
Sand Fine	Percent	10.24	10.58	9.13	21.89	80.69	12.08	6.57
Silt	Percent	77.50	70.29	82.64	2.94	-1.69	-0.14	80.25
Clay	Percent	4.44	4.55	5.01	1.16	1.69	0.14	9.23
Total Organic Carbon								
TOC	mg/kg	95,000	150,000	110,000	4,700	31,000	960	62,000

NOTE:
mg/kg = milligrams per kilogram
N/A = Not applicable
TOC = Total organic carbon

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ATTACHMENT A

PHOTOGRAPH LOG

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Photographic Documentation
Location: Iona Island, New York

Photograph 01: Brush clearing, looking west from DU-22.

Photograph 02: Brush clearing of dump area DUs, looking northwest from Round Island across DU-18 and DU-17

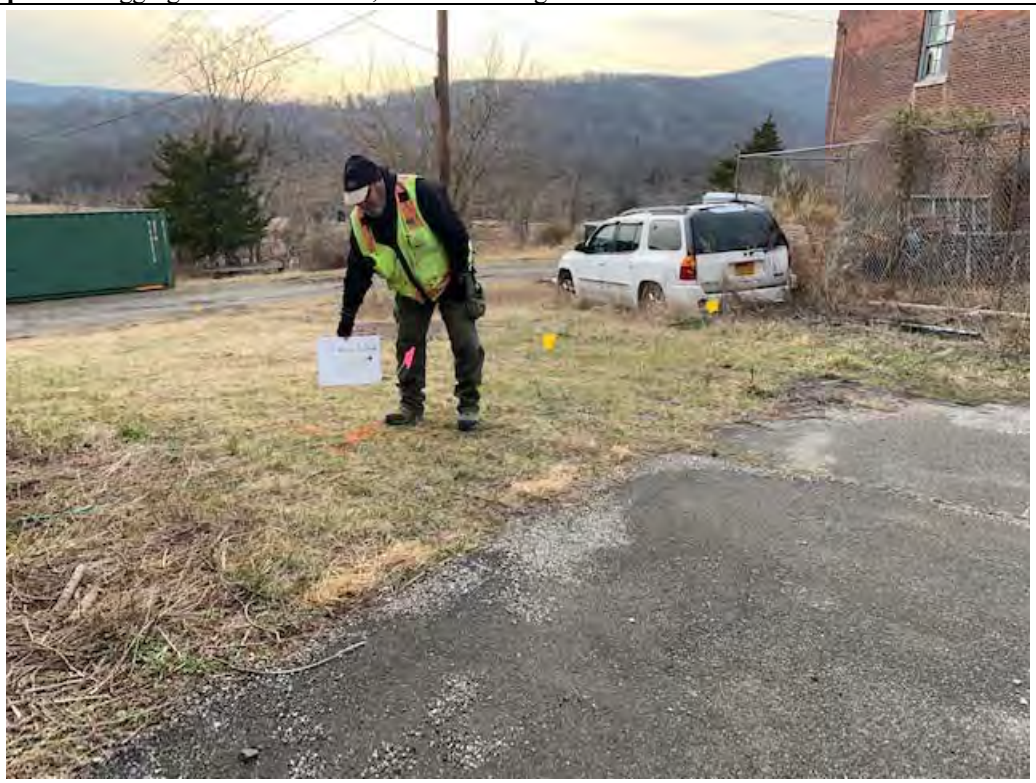
Photographic Documentation
Location: Iona Island, New York

Photograph 03: UXO Clearance Surface Sweep DU-16 looking southwest

Photograph 04: UXO Clearance Dump Area Surface Sweep looking southeast

Photographic Documentation
Location: Iona Island, New York

Photograph 05: Flagging DU Grid Corners, DU-20 looking northeast



Photograph 06: Flagging DU lanes, DU-20 looking west

Photographic Documentation
Location: Iona Island, New York

Photograph 07: Incremental sample collection, DU-9 looking northeast

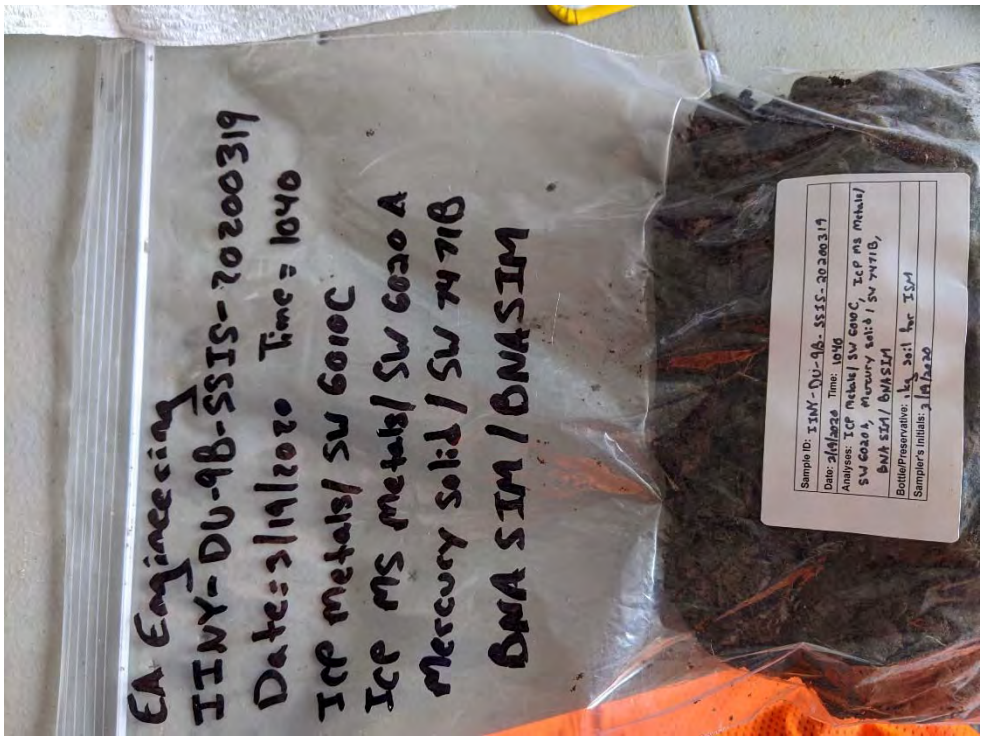
Photograph 08: Incremental sample collection, DU-10 looking northeast

Photographic Documentation
Location: Iona Island, New York

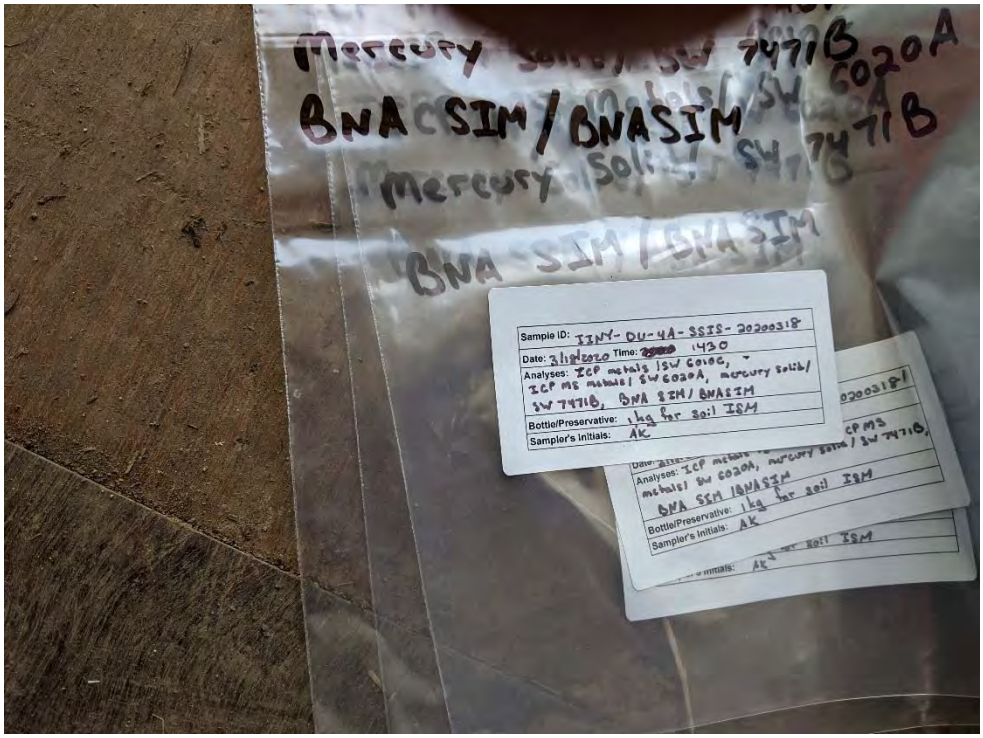
Photograph 09: Subsurface soil sampling at DU-8

Photograph 10: Attempting subsurface soil sampling

Photographic Documentation
Location: Iona Island, New York



Photograph 11: Sample processing and labeling



Photograph 12: Sample processing and labeling

Photographic Documentation
Location: Iona Island, New York

Photograph 13: Exposed bedrock and shallow soil, DU-23 looking east.

Photograph 14: Exposed bedrock and shallow soil, DU-23 looking southeast.

Photographic Documentation
Location: Iona Island, New York

Photograph 15: Looking southwest through center of DU-18. Round Island to southeast.

Photograph 16: Looking southeast across DU-17 and DU-18.

Photographic Documentation
Location: Iona Island, New York

Photograph 17: Exposed bedrock, limited soil looking southeast across DU-24.

Photograph 18: Looking southeast along edge of DU-24. Hudson River shoreline and sediment. Area is within tidal influence of the Hudson River, which is a known PCB source.

Photographic Documentation
Location: Iona Island, New York

Photograph 19: Exposed bedrock, limited soil, looking southwest across DU-24.

Photograph 20: Du-8, looking west toward Building 212.

Photographic Documentation
Location: Iona Island, New York

Photograph 21: DU-8, looking toward south toward Building 222 (area between Building 212 and Building 222)

Photograph 22: Drums in DU-8 between Building 212 and Building 222

Photographic Documentation
Location: Iona Island, New York

Photograph 23: Drums in DU-8 between Building 212 and Building 222.

Photograph 24: Roofing tiles in DU-8 near Building 212.

Photographic Documentation
Location: Iona Island, New York

Photograph 25: Surface conditions in central portion of Iona Island, looking northeast.

Photograph 26: Exposed bedrock and surface conditions in DU-15 looking west/southwest

Photographic Documentation
Location: Iona Island, New York

Photograph 27: Surface conditions looking north/northeast from DU-15

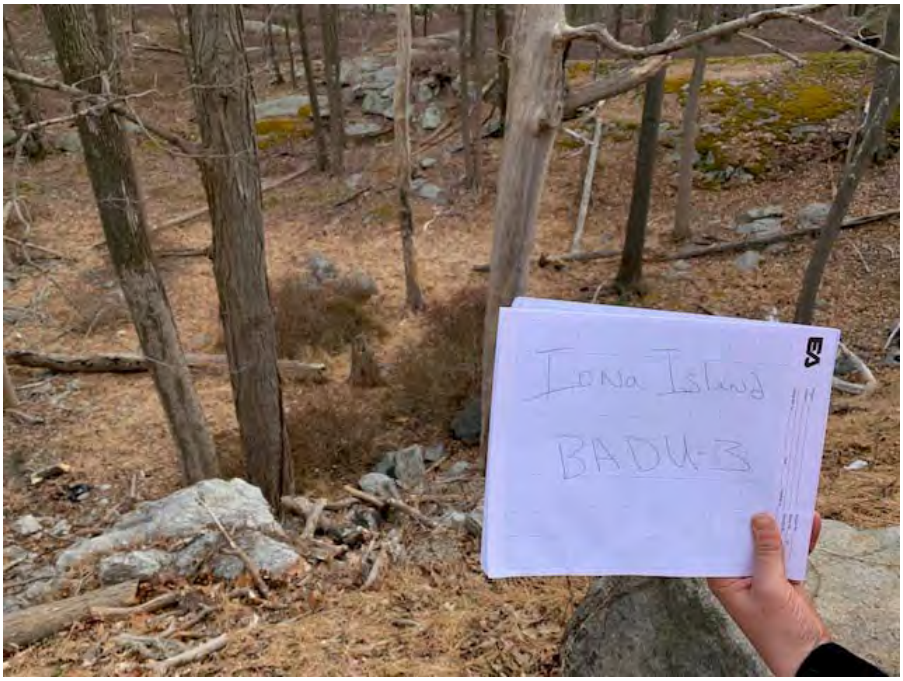
Photograph 28: Surface conditions looking north/northeast from DU-4

Photographic Documentation
Location: Iona Island, New York

Photograph 29: BADU-1 looking north

Photograph 30: BADU-2 looking north

Photographic Documentation
Location: Iona Island, New York



Photograph 31: Surface conditions at BADU-3 looking south

Photograph 32: BADU-4 looking south

Photographic Documentation
Location: Iona Island, New York

Photograph 33: BADU-5 looking north

Photograph 34: BADU-6 looking south

Photographic Documentation
Location: Iona Island, New York

Photograph 35: BADU-7 looking north

Photograph 36: BADU-8 looking south

UFP-QAPP Addendum Appendix B

Iona Island Shoreline Reconnaissance Survey Technical Memorandum

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15 April 2020

TECHNICAL MEMORANDUM

TO: Cheryl Montgomery, USACE ERDC
Erin Kirby, USACE NAE

FROM: Tom King, PWS, EA
Melissa Beauchemin, EA
Tim Reese, EA

CC: Todd Beckwith, USACE NAB
Dan Hinckley, EA
Amanda Kohn, EA

SUBJECT: Iona Island Shoreline Reconnaissance Survey

On 6-7 April 2020, two scientists from EA Engineering, Science, and Technology, Inc., PBC (EA) conducted a Shoreline Reconnaissance Survey at the Iona Island Naval Ammunition Depot, Formerly Used Defense Site. The project site is located in Stony Point, Rockland County, New York (Figure 1). Prior to conducting the survey, four "Recon Areas" were established (Figure 2) in the Hazardous Toxic and Radioactive Waste (HTRW) Remedial Investigation (RI) Uniform Federal Policy (UFP) - Quality Assurance Project Plan (QAPP) for the project site (EA, 2020). These areas include:

- Area 1 – Hudson River Shoreline
- Area 2 – Iona Marsh Shoreline
- Area 3 – Western Shoreline between Iona and Round Island
- Area 4 – CSX Railroad Tracks (West and East side)

In addition to surveying the four identified shoreline Recon Areas, EA personnel also generally characterized the upland conditions throughout Iona and Round Islands and conducted a reconnaissance of potential background sediment sampling locations.

The purpose of the Shoreline Reconnaissance Survey was to identify potential areas of sediment accumulation that could be suitable for sampling along the shoreline, identify and document habitat areas for potential ecological receptors, and identify potential preferential pathways to the shoreline from the upland portion of the island.

Should the results of the Phase I soil sampling investigation on the uplands portion of Iona Island indicate source-pathway-receptor interaction, including a pathway from Iona Island to the shoreline, potential habitat and/or receptors, and if physical parameters indicate a sink of contaminants, then sediments samples will be collected for chemical parameters as part of the Phase II investigation to evaluate potential site impacts to the Iona Island shoreline that are representative of significant habitat, could present a potential exposure pathway to human or ecological receptors, and could potentially be impacted by site-related contaminants.

During the survey, EA personnel traversed the shoreline areas on foot to identify potential areas of interest. EA marked areas of interest with orange and white survey flagging which were also recorded with a sub-meter accurate GPS unit. The areas of interest were geo-referenced on an aerial photograph provided in Appendix A (Figure 3). Photographs of key features are presented in Appendix B and the location of each photograph was mapped on Figure 4.

Area 1 – Hudson River Shoreline

Recon Area 1 consists of the shoreline along the Hudson River, which is located on the east side of Iona Island. The majority of the shoreline in this Recon Area consists of a stone river wall which was measured as approximately 6 feet in height (Photo 5). EA identified approximately 1,100 feet of river wall, although the wall is not contiguous along the shoreline—three large breaks in the wall were identified where the wall has partially collapsed (Photo 6). Additionally, small gaps between the stones were also observed along the length of the wall where the Hudson River passes through the wall at high tide and forms pockets of water behind the wall. EA observed large areas of cobble and boulder-sized stone at the northern and southern ends of the river wall as well as at the identified wall breaks. The large stones were mostly submerged during high tide but exposed during low tide (Photos 1 and 2). EA identified amphipods and aquatic snails on the underside of many of the large stones when flipped over (Photos 3 and 4). However, there was little silty or fine sediment in this area, with the majority of the substrate consisting of cobble and course-grained sand.

The majority of the area along the river wall consists of exposed bedrock (Photo 11) and gravel with the exception of some small pockets of sediment deposition. EA identified three small distinct areas of sediment deposition along the shoreline of Recon Area 1 (Photos 14 and 17). Each of these areas was identified for sediment sampling for grain size and total organic carbon (TOC). Sediment sampling was conducted on 7 April 2020.

EA also identified 6 potential outfall pipes along the river wall area of Recon Area 1. Pipe 1 was identified as an 18-inch concrete pipe located on the northern end of the river wall (Photo 6). This outfall pipe extends approximately 8 feet into the Hudson River during high tide. Pipe 2 was identified as a 12-inch steel pipe located in the central portion of Recon Area 1, near the historic causeway/pier (Photo 8). Additional steel piping and cables were observed in this area. It was unclear during the survey if this 12-inch pipe was part of the causeway road infrastructure (conduit piping) or an outfall discharge pipe. Approximately 30 feet south of Pipe 2, three additional discharge pipes were observed—two 8-inch steel pipes were identified in the wall and one 10-inch terracotta pipe was identified partially exposed in the ground above the wall (Photos 9 and 10). Lastly, Pipe 3 is a 14-inch concrete pipe identified at the southern end of the wall (Photo 12).

The pipes were identified as potential preferential pathways leading to the shoreline although their origins could not be identified at the time of the field survey. Based on the locations of mapped former buildings/structures identified on Figure 2, Pipes 1 and 3 appear to be in the areas of former buildings/structures. Pipe 2 does not appear to be in an area with former buildings/structures.

Area 2 – Iona Marsh Shoreline

Recon Area 2 consists of the shoreline area along the west side of Iona Island. This area is divided by a railroad embankment which supports an active CSX rail line. The western side of the railroad tracks and south of the entrance road consists of a tidal marsh (Photo 23) dominated by cattails (*Typha spp.*); the area east of the railroad tracks (Photo 18) is dominated by an extremely dense monotypic stand of common reed (*Phragmites australis*). The western side of the railroad tracks and north of the entrance road is dominated by phragmites as well. Recon Area 2 consists of typical tidal marsh areas common along the Hudson River. A fine-grained sediment bottom is present throughout the marsh (Photo 30). Three sediment sample locations were selected in this area to document sediment characteristics.

Although no outfall pipes were identified during the recon survey along the shoreline of Recon Area 2, a potential preferential pathway was identified. One groundwater seep was observed on the northwestern portion of Recon Area 2, in the vicinity of the entrance road to Iona Island (Photo 23). This groundwater seep was identified along the upland slope between the marsh and the existing railroad tracks. EA staff observed groundwater discharge along the upland slope into the marsh and a sediment sample was taken at this location. An outfall pipe was not observed in this location.

Area 3 – Western Shoreline Between Iona and Round Island

Recon Area 3 consists of the shoreline area on the southwest side of Iona Island and western portion of Round Island. This area includes the low portion of land between the two islands. Unlike Recon Areas 1 and 2, this area is not a true shoreline, as historic fill and disturbance has significantly altered this area. Recon Area 3 consists of a mix of *Phragmites* tidal wetland on the southern and western end of the area, a tidal wetland swale consisting of various plant species, and a rip-rap filled drainage swale.

Although no outfall pipes were identified during the survey along the shoreline of Recon Area 3, two potential preferential pathways were identified. The southern end of Iona Island appears to drain down towards the *Phragmites* dominated low point between Iona Island and Round Island. This area slopes slightly to the south east to a rip-rap filled drainage swale (Photo 20) that directs flow to the south, towards the tidal marsh associated with Recon Area 2. A tidal wetland swale also identified along Recon Area 3 appears to convey flow to the west toward the tidal marsh associated with Recon Area 2.

Just north of Recon Area 3, EA identified a *Phragmites* filled wetland depression on the north side of the Iona Island perimeter road. This depression appears to collect surface runoff from Iona Island, but EA staff were unable to identify a culvert beneath the road connecting this depression to the tidal wetland swale within Recon Area 3.

Area 4 – CSX Railroad Tracks (West and East Side)

Recon Area 4 includes the areas along the east and west side of the railroad track embankment and primarily consists of rip-rap along an upland embankment. The northern portion of Recon Area 4 includes a scrub-shrub upland along the edge of the rip-rap (Photo 24); the southern end of the recon area includes a transition from rip-rap to existing marsh areas associated with Recon Area 2 (Photo 19).

EA did not identify any discharge outfall pipes along Recon Area 4. No culverts were identified beneath the railroad embankment that could connect drainage from Iona Island on the east of the railroad embankment to the marsh on the western side of the railroad embankment. EA identified only one hydrological connection through the railroad embankment, connecting the cattail marsh to the west with the *Phragmites* marsh on the east. This connection is located outside the project area at an embankment opening (i.e., shallow bridge) approximately 1,500 feet away, across the marsh from Round Island.

Flora and Fauna Observations

In addition to identifying potential pathways and characterizing the shoreline conditions of the site, EA's environmental scientists also documented wildlife to determine potential ecological receptors at the site. Wildlife observations included incidental observations and did not include a formal survey for specific species. In general, the observations made are consistent of what would be expected for the habitat areas described above. A list of wildlife observations along with the general recon area depicting where each species was identified is provided in Table 1.

Table 1. Wildlife Observations

Common Name	Scientific Name	Recon Area Observed				
		Area 1	Area 2	Area 3	Area 4	Upland
White tailed deer	*Odocoileus virginianus*		X	X		X
Grey squirrel	*Sciurus carolinensis*				X	X
Northern water snake	*Nerodia sipedon*			X		
Black rat snake	*Panthrophis obsoletus*		X	X		
Painted turtle	*Chrysemys picta*		X			
Terrestrial snail	--	X				
Aquatic snail	--	X				
Amphipod	--	X				
Turkey vulture	*Cathartes aura*	X	X	X	X	X
Osprey	*Pandion haliaethus*	X	X	X		X
American crow	*Corvus brachyrhynchos*	X			X	X
Red-winged blackbird	*Agelaius phoeniceus*		X	X		
Mourning dove	*Agelaius phoeniceus*					X
Northern cardinal	*Cardinalis cardinalis*					X
Northern bluejay	*Cyanocitta cristata*			X		X
Great scaup	*Aythya marila*	X				
Pileated woodpecker	*Dryocopus pileatus*					X
Ring-billed gull	*Larus delawarensis*	X				X

As previously mentioned, *Phragmites* and cattail were the dominant aquatic vegetation. The upland habitat consists primarily of invasive species including Japanese barberry (*Berberis thunbergii*), multiflora rose (*Rosa multiflora*), wineberry (*Rubus phoenicolasius*), and Japanese honeysuckle (*Lonicera japonica*). Other dominant species located within the upland areas included maples (*Acer spp.*), *Forsythia spp*., devil's walkingstick (*Aralia spinosa*), and red cedar (*Juniperus virginiana*). Milkweed (*Asclepias syriaca*) was also identified.

Sediment Sampling

EA collected sediment samples on 7 April 2020 from areas identified as depositional. EA identified seven sediment sampling locations, including three locations in Recon Area 1 (SD 04, SD 05, and SD 06), three locations in Recon Area 2 (SD 01, SD 02, SD 03), and one location in Recon Area 3 (SD 07). The seven sediment locations are depicted on Figure 3. Sediment samples were collected for analysis of grain size and TOC. Grain size appears to vary from coarse sand and cobbles in Recon Area 1 to fine silts and clays in Recon Areas 2 and 3.

Potential Sediment Background Locations

EA identified two potential upstream background sediment sampling locations (Figure 5). The first potential background sampling location was identified as Constitution Island, which is located approximately 7 miles upstream of Iona Island on the eastern bank of the Hudson River. The island consists of approximately 175 acres of upland habitat and another 240 acres of marsh, similar to that of Iona Island. A gate prevented access to the island and signs indicated it is government property with restricted access and is believed to be part of West Point Military Academy.

The second potential background location was identified as Con Hook, which is a peninsula located approximately 3 miles upstream of Iona Island on the western side of the Hudson River. It consists of approximately 7 acres of upland

habitat and another 12 acres of a *Phragmites*-dominated marsh land, similar to that of Iona Island. Access appears only possible via boat. A boat ramp is located approximately 2 miles south of Con Hook in Mine Dock Park.

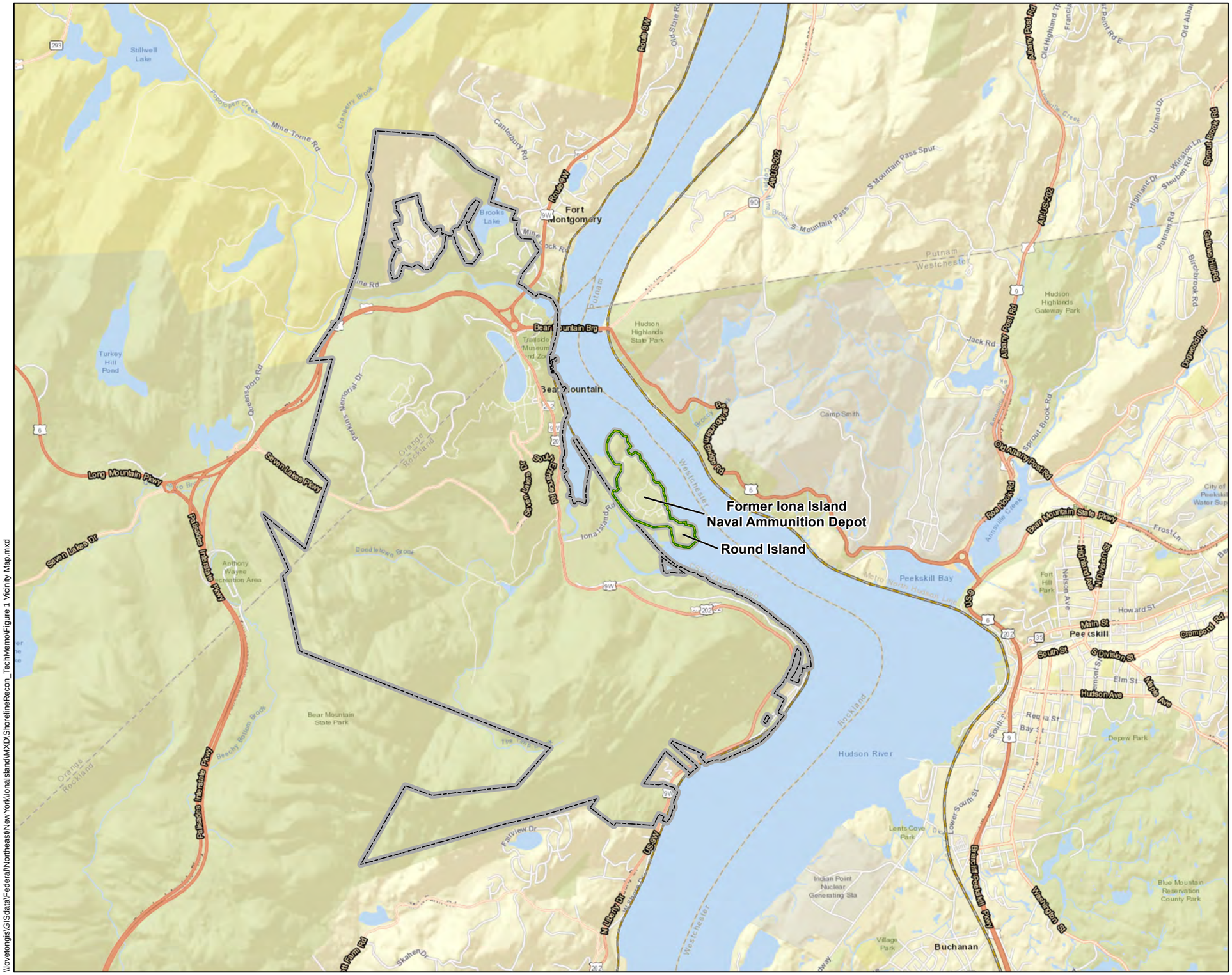
Reference

EA Engineering, Science, and Technology Inc., PBC (EA). 2020. *Uniform Federal Policy -Quality Assurance Project Plan, Hazardous Toxic and Radioactive Waste Remedial Investigation, Iona Island Naval Ammunition Depot* - Formerly Used Defense Site. January 2020.

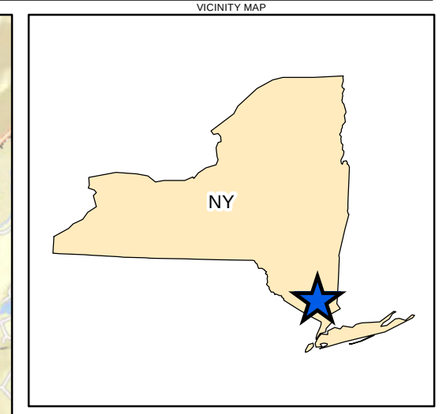
Appendix

A	Figures
B	Site Photographs

APPENDIX A
FIGURES



VICINITY MAP



Legend

- FUDS Boundary
- Bear Mountain State Park Boundary

0 0.5 1
Miles
1 in = 1 miles

N

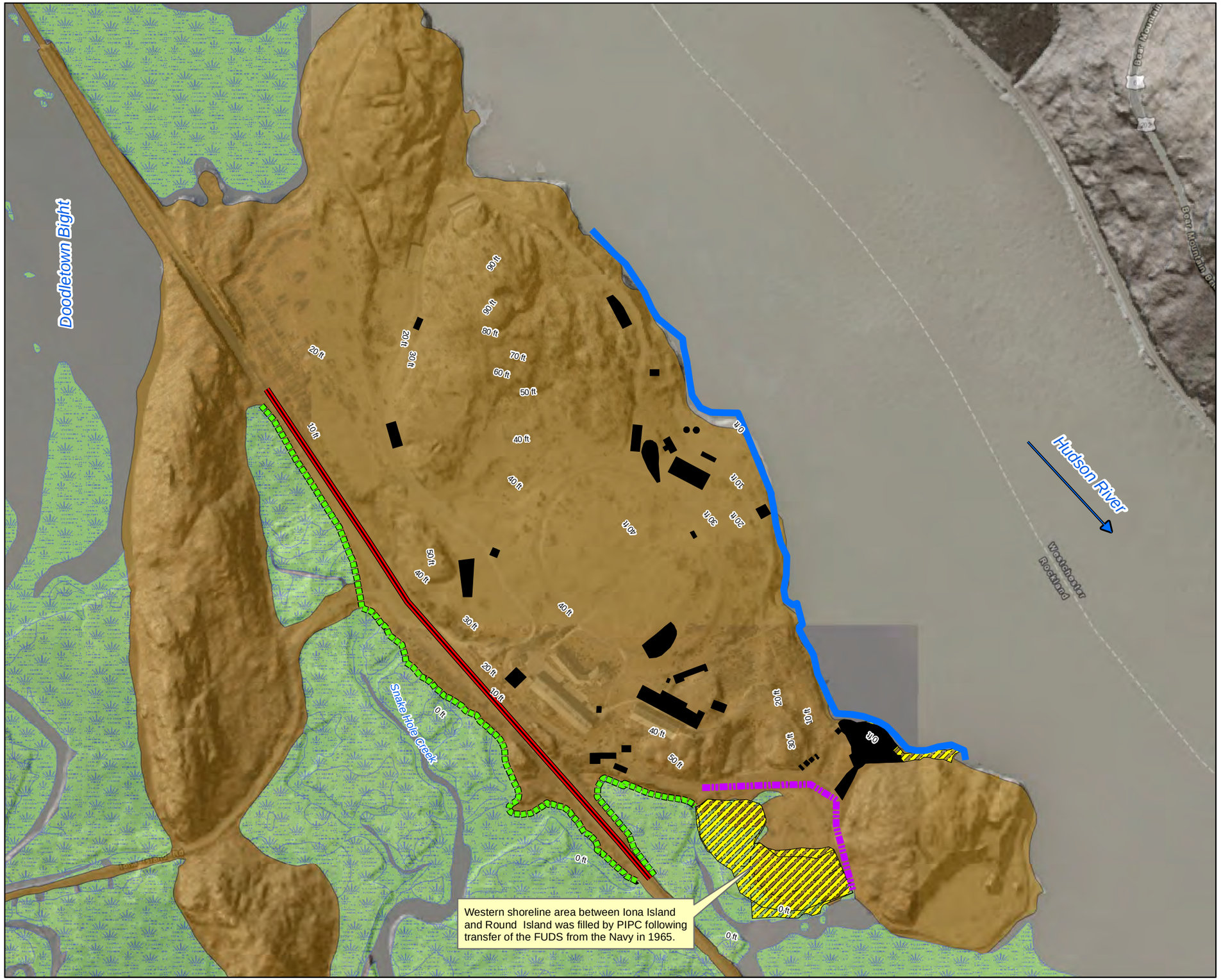
FIGURE 1
Vicinity Map

IONA ISLAND NAVAL AMMUNITION DEPOT FUDS
ROCKLAND COUNTY, NY

Aerial: ESRI ArcGIS Online Map Service

Map Date: 4/11/2020

\\lovetongis\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\ShorelineRecon_TechMemo\Figure 1 Vicinity Map.mxd



VICINITY MAP

Legend

- HTRW RI Footprints of Former Buildings/Structures
- Disturbed Ground/Fill Area
- Upland
- Marsh/Wetland

Phase I Shoreline Reconnaissance Areas

- Recon Area 1 Hudson River Shoreline
- Recon Area 2 Iona Marsh Shoreline
- Recon Area 3 Western Shoreline Between Iona and Round Island
- Recon Area 4 CSX Railroad Tracks (East and West Sides)

0 400 800
Feet
1 in = 400 ft

N

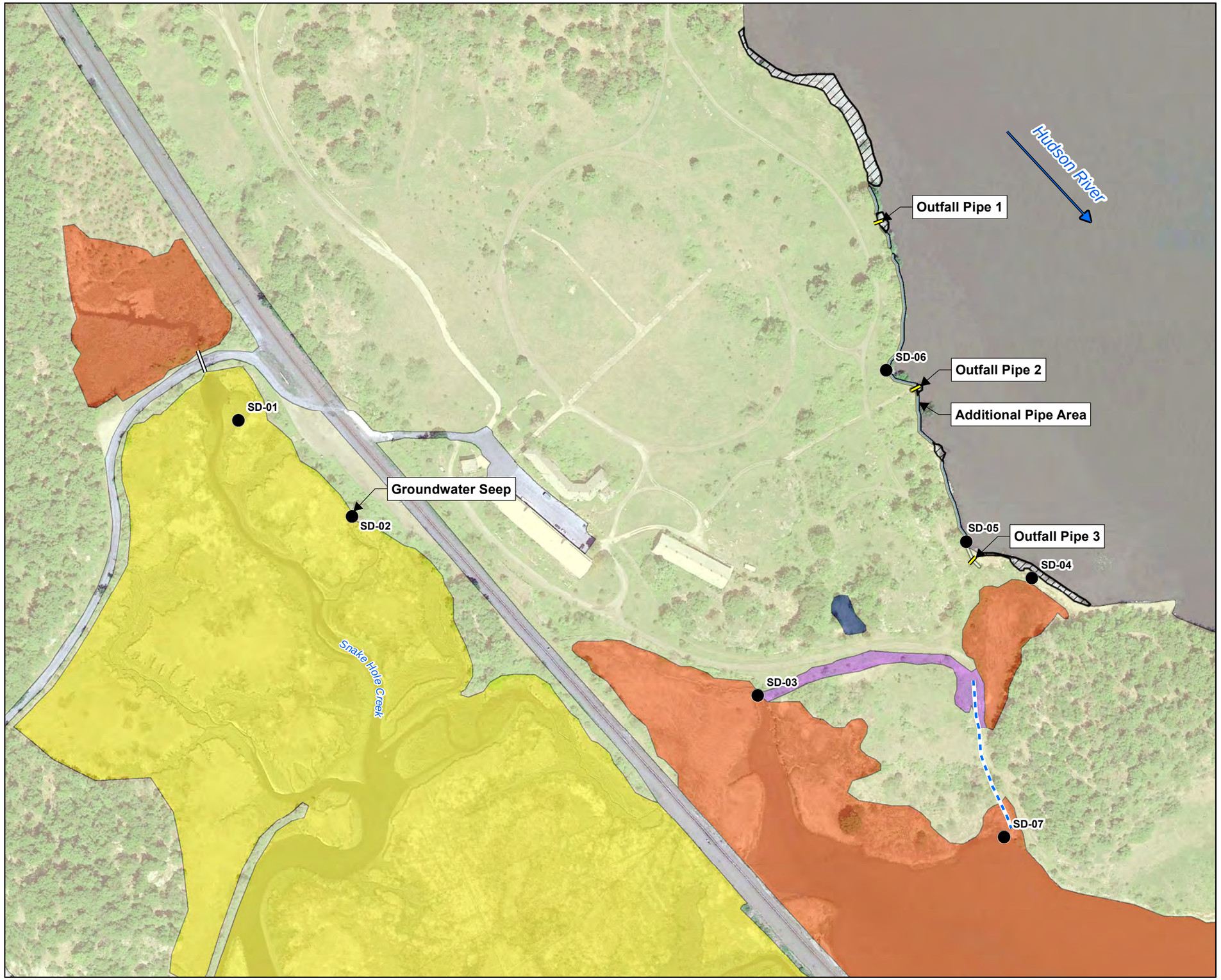
FIGURE 2
Phase I Shoreline Reconnaissance Survey Areas

IONA ISLAND NAVAL AMMUNITION DEPOT FUDS
ROCKLAND COUNTY, NY

Aerial: ESRI ArcGIS Online Map Service

Map Date: 4/11/2020

\\lovetongis\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\ShorelineRecon_TechMemo\Figure 2 Shoreline Recon Survey Areas.mxd



VICINITY MAP

Legend

- Sediment Sample Location
- Potential Preferential Pathway
- Culvert
- Outfall Pipe
- River Wall
- Upland
- Phragmites Wetland (Nontidal)
- Phragmites Tidal Wetland
- Cattail Tidal Wetland
- Tidal Wetland Swale
- Macroinvertebrate Observation Areas (Cobble and large gravel)

0 250 500
Feet
1 in = 250 ft

N

FIGURE 3
Phase I Shoreline Reconnaissance Observations

IONA ISLAND NAVAL AMMUNITION DEPOT FUDS
ROCKLAND COUNTY, NY

Aerial: Google Earth Aerial 2018

Map Date: 4/13/2020

\\lovetongis\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\ShorelineRecon_TechMemo\Figure 3 Shoreline Recon Observations.mxd

VICINITY MAP

Legend

- Photo Location
- Photo Location Direction

0 250 500
Feet
1 in = 250 ft

N

FIGURE 4
Photo Location Map

IONA ISLAND NAVAL AMMUNITION DEPOT FUDS
ROCKLAND COUNTY, NY

Aerial: Google Earth Aerial 2018

Map Date: 4/13/2020

\\lovetongis\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\ShorelineRecon_TechMemo\Figure 4 Photograph Location Map.mxd

VICINITY MAP

Legend

Possible Sediment Background Sample Location

FUDS Boundary

FIGURE 5
Possible Sediment Background Sample Locations
IONA ISLAND NAVAL AMMUNITION DEPOT FUDS
ROCKLAND COUNTY, NY

Map Date: 4/13/2020
Source: ESRI Aerial 2018
Projection: NAD 1983 State Plane New York East US Foot

\\lovetongis\GISdata\Federal\Northeast\NewYork\IonaIsland\MXD\ShorelineRecon_TechMemo\Figure 5 Possible Sediment Background Sample Locs.mxd evan

APPENDIX B
SITE PHOTOGRAPHS

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

1. Northernmost portion of Recon Area 1 at high tide, before start of river wall.

2. Northernmost portion of Recon Area 1 at low tide.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

3. Amphipod observed under shoreline rocks during low tide.

4. Snail observed under shoreline rocks during low tide.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

5. River wall located along the majority of Recon Area 1.

6. Pipe #1 identified along the northern portion of Recon Area 1.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

7. Sediment area observed at low tide at sample area SD-06.

8. Pipe #2 observed along Recon Area 1.

Photographic Record

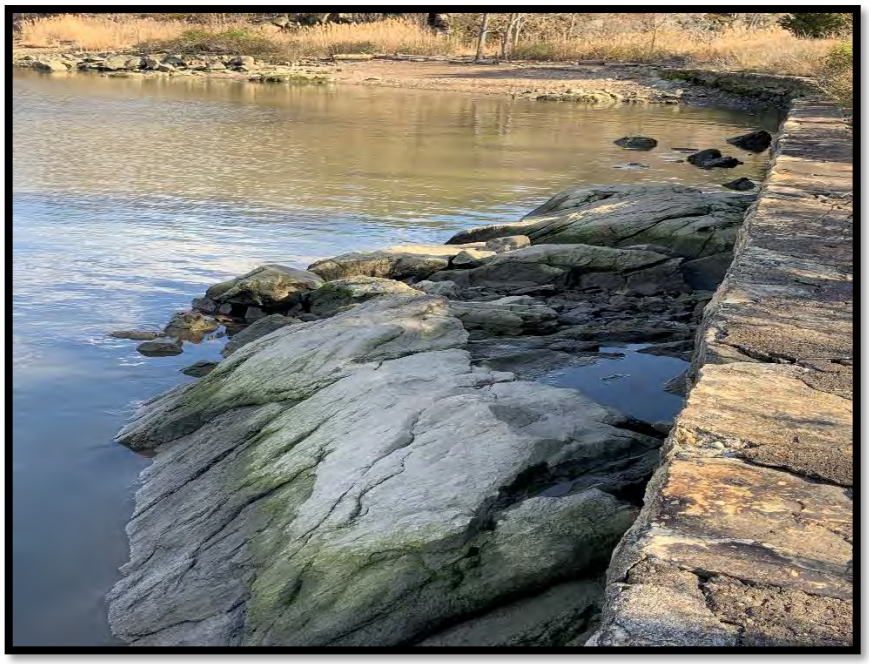
Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

9. Terracotta pipe identified partially buried near the historic causeway in Recon Area 1.

10. Two additional metal pipes identified along the river wall, near the historic causeway in Recon Area 1.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020



11. Sample location SD-05 located between the exposed bedrock and beach area in Recon Area 1.

12. Pipe #3 located along the river wall in Recon Area 1.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

13. Coarse sand, beach glass and pebble material along the beach area on the southern portion of Recon Area 1.

14. Southernmost portion of Recon Area 1 at high tide at sediment location SD-04.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

15. Southernmost portion of Recon Area 1 at low tide.

16. Water depth of approximately 5 feet along the river wall in Recon Area 1 at high tide.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

17. Northern shoreline of Round Island.

18. *Phragmites* dominated marsh in Recon Area 2, on the eastern side of railroad tracks.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

19. Rip-rap slope and water depth of three feet along the eastern side of railroad tracks, across from Round Island.

20. Potential pathway located between Round Island and the existing fill area.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

21. View of Recon Area 3 from Round Island.

22. View of *Phragmites* dominated marsh between Iona Island and Round Island.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

23. Potential groundwater seep area along Recon Area 2 (Location of SD 02).

24. Typical view of railroad tracks in Recon Area 4.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

25. Top of river wall with evidence of historic railing or fence posts.

26. Typical metal piping observed in Recon Areas 1 and 3 which appear to be from historic railing or fence structure.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

27. Observed fence and wall drainpipe identified on Round Island.

28. Sediment sample location SD-01.

Photographic Record

Shoreline Reconnaissance Photographs – Iona Island
6-7 April 2020

29. Sediment Sample Location SD-03 at hightide.

30. Sediment sample location SD-07 at low tide.

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UFP-QAPP Addendum Appendix C

Laboratory Certifications

- Eurofins TestAmerica Denver DoD ELAP DOECAP
- Eurofins-Lancaster DoD ELAP Certification
- Katahdin Analytical Services, LLC DoD ELAP Certification

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SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Roxanne Sullivan Phone: 303-736-0100
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2021 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Boron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Chromium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Lead	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lithium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Magnesium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Manganese	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Selenium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silica	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silicon	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silver	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Strontium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Thallium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thorium	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Tin	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Titanium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Tungsten	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Uranium	EPA 200.8	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nutrients			
Nitrate (as N)	By calculation	EPA 300.0 EPA 9056/9056A By Calculation/Nitrate by Calc	EPA 9056/9056A By Calculation/Nitrate by Calc
Nitrate-nitrite (as N)	EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Orthophosphate (as P)	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	EPA 365.1	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinty)	SM 2320B-1997	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Flashpoint	----------	EPA 1010A	----------
Hexavalent chromium	----------	EPA 7196A	EPA 7196A
pH	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	EPA 9071B
Percent moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, total	----------	----------	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Sulfide, Total	SM 4500S2 D	EPA 9034/SM 4500S2 D	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2
Purgeable Organics (Volatiles)			
Acetone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acetonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Acrolein	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acrylonitrile	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Allyl Chloride	----------	EPA 8260B/8260C	EPA 8260B/8260C
Benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C/ AK101/OK DEQ GRO
Benzyl Chloride	----------	EPA 8260B/8260C/8260C	EPA 8260B/8260C/8260C

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Bromobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromodichloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromoform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Butadiene	----------	----------	----------
2-Butanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
n-Butyl alcohol	----------	EPA 8260B/8260C EPA 8015B/8015C	EPA 8260B/8260C EPA 8015B/8015C
tert-Butyl alcohol (2-Methyl-2-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
sec-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
tert-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Carbon disulfide	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Carbon tetrachloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloro-1,3-butadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloroethyl vinyl ether	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1-Chlorohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroprene	----------	EPA 8260B/8260C	EPA 8260B/8260C
4-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dibromochloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
Dibromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dibromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2 Dibromoethane (EDB)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,2-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Dichlorofluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloropropane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2,2-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloropropene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Diethyl ether	----------	EPA 8260B/8260C	EPA 8260B/8260C
Di-isopropylether	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dioxane	EPA 624/624.1	EPA 8260B/8260C EPA 8260B/8260C SIM	EPA 8260B/8260C EPA 8260B/8260C SIM
Ethanol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Ethyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl tert-butyl ether	----------	----------	----------
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx
Hexane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Hexanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Hexachlorobutadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropyl alcohol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Isopropyltoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Iodomethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methacrylonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methylene chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Methyl ethyl ketone (MEK)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl isobutyl ketone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl tert-butyl ether (MtBE)	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
4-Methyl-2-pentanone (MIBK)	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Naphthalene	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
2-Nitropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Pentachloroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Pentanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Propionitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Propylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Styrene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
1,1,1,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrachloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrahydrofuran	----------	EPA 8260B/8260C	EPA 8260B/8260C
Toluene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1,1-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichlorofluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichloropropane	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl acetate	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Xylenes, Total	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
1,2-Xylene (o-Xylene)	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
m+p-Xylene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/ K DEQ GRO
Methane	----------	RSK-175	----------
Ethane	----------	RSK-175	----------
Ethylene (Ethene)	----------	RSK-175	----------
Acetylene	----------	RSK-175	----------
Acetylene ethane	----------	RSK-175	----------
Extractable Organics (Semivolatiles)			
Acenaphthene	EPA 625/625.1/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acenaphthylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acetophenone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alachlor	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Aramite	----------	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde	----------	EPA 8270C 8270D	EPA 8270C/8270D
Benzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzo(a)anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(b)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(k)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(ghi)perylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(a)pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzyl alcohol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,1-Biphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethoxy) methane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethyl) ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroisopropyl) ether (2,2'Oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Ethylhexyl) phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
butyl Benzyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Caprolactam	----------	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-chloro-3-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Cresols	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diallate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,h) anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Dibenzofuran	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
2,4-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D	EPA 8270C/8270D
alpha-, alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-butyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-octyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4,6-Dinitro-2-methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dinoseb	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dioxane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx
Ethyl Methanesulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	----------	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	EPA 625/625.1	EPA 8270C/8270D EPA8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Fluorene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Hexachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorophene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Isodrin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	----------	EPA 8270C/8270D	EPA 8270 C/8270D
Methapyrilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-methyl-4,6-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
methyl Methane sulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
1,4-Naphthoquinone	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
5-nitro-o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-chloropropane)	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, methyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	EPA 625/625.1	EPA 8270C/8270D EPA 8321A/8321B	EPA 8270C/8270D EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Phenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
p-Phenylene Diamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Phorate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Pyridine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Safrole	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Sulfotepp	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin	----------	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Tribromophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Tributyl phosphate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Pesticides/Herbicides/PCBs			
Aldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
beta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4-D	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dalapon	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
2,4-DB	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
4,4'-DDD	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Dieldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb	----------	EPA 8151A EPA 8321A	EPA 8321A
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608/608.3	EPA 8081A /8081B	EPA 8081A/8081B
Endonsulfan sulfate	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
MCPP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA8321A
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Total PCBs	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Picrolam	----------	EPA 8151A	EPA 8151A
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Toxaphene	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-Triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
DNX	----------	EPA 8330A/8330B	EPA 8330A/8330B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
MNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
TNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Diamino-4-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Diamino-6-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	SM 2510B	EPA 9050A	EPA 9050A
Corrosivity	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
Turbidity	EPA 180.1	----------	----------
Organic Prep Methods			
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Soxhlet extraction	----------	----------	EPA 3540C
Microwave extraction	----------	----------	EPA 3546
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Waste dilution	----------	EPA 3580A	EPA 3580A
Solid phase extraction	----------	EPA 3535A	----------
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Organic Cleanup Procedures			
Florisil cleanup	----------	EPA 3620B	EPA 3620B
Florisil cleanup	----------	EPA 3620C	EPA 3620C
Sulfur cleanup	----------	EPA 3660A	EPA 3660A
Sulfuric acid/Permanganate cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid digestion total recoverable or dissolved metals	----------	EPA 3005A	----------
Acid digestion for total metals	----------	EPA 3010A	----------
Acid digestion for total metals	----------	EPA 3020A	----------
Acid digestion of sediments, sludges and soils	----------	----------	EPA 3050B

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	**Method(s)**
Metals	
Cadmium	EPA 6010C/6010D
Chromium	EPA 6010C/6010D
Lead	EPA 6010C/6010D
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (Volatiles)	
tert-Amyl Methyl Ether	EPA 8260B/8260C
Benzene	EPA 8260B/8260C
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B/8260C
1,2-Dichloroethane	EPA 8260B/8260C
Di-isopropylether	EPA 8260B/8260C
Ethyl benzene	EPA 8260B/8260C
Ethyl tert-butyl ether	EPA 8260B/8260C
Gas Range Organics (GRO)	EPA 8015B/8015C/8015D

Parameter/Analyte	**Method(s)**
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C
Naphthalene	EPA 8260B/8260C
Toluene	EPA 8260B/8260C
Xylenes, total	EPA 8260B/8260C
1,2-Xylene	EPA 8260B/8260C
M+P-Xylene	EPA 8260B/8260C
Extractable Organics (Semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D (WY: C10-C32)
Organic Prep Methods	
Volatiles purge and trap	EPA 5030B (water) /5030A (solids)

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Accredited Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 12th day of August 2019.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2021

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL LLC
2425 New Holland Pike
Lancaster, PA 17601
Dorothy M. Love Phone: 717-556-7327

ENVIRONMENTAL

Valid To: November 30, 2022 Certificate Number: 0001.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Demands				
COD	----------	EPA 410.4	----------	----------
Total Organic Carbon	----------	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310 B-2011
Anions				
Ammonia	----------	EPA 350.1	EPA 350.1	SM 4500-NH3 B/C-2011
Fluoride	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrate (as N)	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrite (as N)	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0

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Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Bromide	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Chloride	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Sulfate	----------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Wet Chemistry				
Alkalinity	----------	SM 2320B-2011	----------	----------
Corrosivity	----------	----------	SW-846 Chapter 7	SW-846 Chapter 7
Cyanide	----------	EPA 9012B	EPA 9012B	EPA 9012B
Filterable Residue (TDS)	----------	SM 2540C-2011	----------	----------
Flashpoint	----------	----------	EPA1010A	EPA 1010A
Grain Size	----------	----------	----------	ASTM D422
Hexavalent Chromium Digestion	----------	----------	----------	EPA 3060A
Hexavalent Chromium	----------	EPA 7196A EPA 7199	EPA 7196A EPA 7199	EPA 7196A EPA 7199
Ignitability	----------	----------	40 CFR 261.21	40 CFR 261.21
Nitrate/Nitrite	----------	----------	EPA 353.2	----------
Non-filterable Residue (TSS)	----------	SM 2540D-2011	----------	----------
pH	----------	SM 4500 H+B-2011 EPA 9040B EPA 9040C	EPA 9040B EPA 9040C	EPA 9045C EPA 9045D
Phenol	----------	EPA 9066	EPA 9066	----------
Reactivity	----------	----------	SW-846 Chapter 7.3	SW-846 Chapter 7.3
Sulfide	----------	EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011	----------	----------
Total Residue	----------	SM 2540B-2011	----------	SM 2540G-2011
Metals				
Metals Digestion	----------	EPA 3005A EPA 3010A EPA 3020A	EPA 3010A EPA 3020A	EPA 3050B
Aluminum	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Antimony	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Arsenic	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Barium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Beryllium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Boron	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Cadmium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Calcium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Chromium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Cobalt	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Copper	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Iron	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lead	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lithium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Molybdenum	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Magnesium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Manganese	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Mercury	----------	EPA 7470A	EPA 7470A	EPA 7471A EPA 7471B
Nickel	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Potassium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Selenium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Silicon	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Silver	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sodium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Strontium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sulfur	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Thallium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Thorium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tin	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Titanium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tungsten	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Uranium	----------	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B
Vanadium	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zinc	----------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zirconium	----------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Purgeable Organics (Volatiles)				
Volatile Preparation	----------	EPA 5030C	EPA 5030C	EPA 5035A
Acetone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acetonitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrolein	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrylonitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Allyl chloride	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Methyl Ether	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Formate	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Benzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromodichloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromoform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Butanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
sec-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon disulfide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon tetrachloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloro-1,3-butadiene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroacetonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1-Chlorobutane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorodifluoromethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloroethyl Vinyl Ether	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Drinking Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
1-Chlorohexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexanone	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Di-Isopropyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dibromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromo-3-chloropropane	EPA 524.2	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
Dibromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromoethane (EDB)	----------	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
1,2-Dichlorobenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,4-dichloro-2-butene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorodi-fluoromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorofluoromethane	EPA 524.2	----------	----------	----------
1,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloropropene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,3-Dichloropropene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,3-Dichloropropene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dioxane	----------	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM
Ethanol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Freon-113	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)]	----------	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101
Heptane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Hexanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Hexachlorobutadiene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isobutyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropyl Alcohol	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Isopropyltoluene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylacrylonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acetate		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Iodide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Ethyl ketone	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylene Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Isobutyl Ketone	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Methyl-2-pentanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylcyclohexane		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Nitropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Naphthalene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Pentachloroethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Propionitrile	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Propylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Styrene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tert-Amyl Ethyl Ether		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrachloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrahydrofuran	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Toluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Trichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Trichlorofluoromethane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichloropropane	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trimethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3,5-Trimethylbenzene	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Vinyl Acetate	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Vinyl Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Xylenes, Total	----------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Xylene (o-Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3+1,4-Xylene (m+p Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Extractable Organics (Semivolatiles)				
Acenaphthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acenaphthylene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acetophenone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Acetylaminofluorene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Alkylated PAHs	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
4-Aminobiphenyl	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Amino-4,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
4-Amino-2,6-dinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Aniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Atrazine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzaldehyde	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzoic acid	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzo (a) anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (b) fluoranthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (k) fluoranthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (ghi) perylene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (a) pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (e) pyrene	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Benzyl Alcohol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Biphenyl	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Chloroethoxy) Methane	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Chloroethyl) Ether	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
bis (2-Chloroisopropyl) Ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Ethylhexyl) Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
4-Bromophenylphenyl Ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Butyl benzyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Caprolactam	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbazole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
4-Chloroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chloro-3-methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chlorobenzilate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Chloronaphthalene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chloronaphthalene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chlorophenyl phenyl ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chrysene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Cresols (Methyl phenols)	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
cis-/trans-Diallate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Diamino-6-nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
2,6-Diamino-4-nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Dibenzo (a,h) acridine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dibenzo (a,h) anthracene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Dibenzofuran	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,2-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,4-Dichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,3-Dichlorobenzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)]	----------	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103
2,4-Dichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,6-Dichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diethyl Phthalate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dimethoate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
p-Dimethylaminoazobenze	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
7,12-Dimethylbenz (a) anthracene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dimethylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dimethyl Phthalate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,3'-Dimethylbenzidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Di-n-butyl Phthalate	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Di-n-octyl phthalate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B	EPA 8330B
1,3-Dinitrobenzene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
1,4-Dinitrobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrotoluene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
2,6-Dinitrotoluene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
1,4-Dioxane	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Diphenylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diphenyl ether	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2-Diphenylhydrazine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Ethyl Methane Sulfonate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Fluoroanthene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Fluorene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobenzene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobutadiene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachlorocyclo-pentadiene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloroethane	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloropropene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	----------	EPA 8330B	EPA 8330B	EPA 8330B
Indeno (1,2,3-cd) Pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Isodrin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Isophorone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Isosafrole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Methycholanthrene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Methyl-4,6-dinitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Methyl methane sulfonate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Methylnaphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylnaphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Methylphenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Naphthalene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,4-Naphthoquinone	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Naphthylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Naphthylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroquinoline-1-oxide	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroaniline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Nitrobenzene	----------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
Nitroglycerin	----------	EPA 8330B	EPA 8330B	EPA 8330B
2-Nitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitrophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
3-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
4-Nitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
5-Nitro-o-toluidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitroso-di-n-butylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiethylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodimethylamine	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
n-Nitrosodimethylethylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosomorpholine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodi-n-propylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiphenylamine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopiperidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopyrrolidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	----------	EPA 8330B	EPA 8330B	EPA 8330B

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
2,2-Oxybis (1-chloropropane)	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachloronitrobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentaerythritol Tetranitrate (PETN)	----------	EPA 8330B	EPA 8330B	EPA 8330B
Perylene	----------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Phenacetin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Phenanthrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Phenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Picoline	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pronamide	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pyrene	----------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Pyridine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Safrole	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4,5- Tetrachlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,3,4,6-Tetrachlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethyl dithiopyrophosphate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethy lead	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetryl	----------	EPA 8330B	EPA 8330B	EPA 8330B
Thionazin	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
o-Toluidine	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4-Trichlorobenzene	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3,5-Trinitrobenzene	----------	EPA 8330B	EPA 8330B	EPA 8330B
2,4,5-Trichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trichlorophenol	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
O,O,O-Tri-ethylphosphorothioate	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trinitrotoluene	----------	EPA 8330B	EPA 8330B	EPA 8330B
Organochlorine Pesticides				
Aldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
beta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
delta-BHC	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-BHC (Lindane)	----------	EPA 8081B	EPA 8081B	EPA 8081B

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
alpha-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Chlordane (Technical)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Chlorobenzilate	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
2,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDD	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDE	----------	EPA 8081B	EPA 8081B	EPA 8081B
4,4'-DDT	----------	EPA 8081B	EPA 8081B	EPA 8081B
Diallate	----------	EPA 8081B	EPA 8081B	EPA 8081B
1.2-Dibromo-3-chloropropane (DBCP)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Dieldrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Dinoseb	----------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Endosulfan I (alpha)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan II (beta)	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan Sulfate	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Aldehyde	----------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Ketone	----------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-Chlordane	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor Epoxide	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorobenzene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorocyclopentadiene	----------	EPA 8081B	EPA 8081B	EPA 8081B
Isodrin	----------	EPA 8081B	EPA 8081B	EPA 8081B
Methoxychlor	----------	EPA 8081B	EPA 8081B	EPA 8081B
Mirex	----------	EPA 8081B	EPA 8081B	EPA 8081B
Toxaphene	----------	EPA 8081B	EPA 8081B	EPA 8081B
PCBs (Aroclors)				
PCB-1016 (Arochlor)	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1221	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1232	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1242	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1248	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1254	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1260	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1262	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1268	----------	EPA 8082A	EPA 8082A	EPA 8082A
PCB congeners (209)	----------	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Herbicides				
2,4,5-T	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4,5-TP (Silvex)	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-D	----------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dalapon	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dicamba	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dichlorprop	----------	EPA 8151A	EPA 8151A	EPA 8151A
Dinoseb	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPA	----------	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	----------	EPA 8151A	EPA 8151A	EPA 8151A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A	EPA 8151A
PCB Homologues				
Monochlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Trichlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Tetrachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Pentachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Hexachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Heptachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Octachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Nonachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Decachlorobiphenyls	----------	EPA 680	EPA 680	EPA 680
Dioxins/Furans				
2,3,7,8-TCDD	EPA 1613B	EPA 8290A	EPA 8290A	EPA 8290A
2,3,7,8-TCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,7,8-PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,6,7,8-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,9-HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
OCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
OCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total PeCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Total PeCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDD	----------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDF	----------	EPA 8290A	EPA 8290A	EPA 8290A
Misc. Headspace Analysis				
Carbon dioxide	----------	RSK-175	RSK-175	----------
Ethane	----------	RSK-175	RSK-175	----------
Ethene	----------	RSK-175	RSK-175	----------
Methane	----------	RSK-175	RSK-175	----------
Hazardous Waste Characteristics				
Toxicity Characteristic Leaching Procedure	----------	----------	EPA 1311	EPA 1311
Synthetic Precipitation Leaching Procedure	----------	----------	EPA 1312	EPA 1312
ASTM Leaching Procedure	----------	----------	ASTM D3987-85	ASTM D3987-85
Other				
Perchlorate	----------	EPA 6850	EPA 6850	EPA 6850
Hydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Formaldehyde	----------	----------	EPA 8315A	EPA 8315A
Methylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	----------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction/Cleanup	----------	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3546 EPA 3550C EPA 3660B, 3620C, 3665A, 3640A

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl Perfluorooctane-Sulfonamidoacetic Acid (NetFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
N-methyl Perfluoroctane-Sulfonamidoacetic Acid (NMeFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorobutanesulfonic Acid (PFBS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorodecanoic Acid (PFDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Perfluorododecanoic Acid (PFDoDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroheptanoic Acid (PFHpA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexanesulfonic Acid (PFHxS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexanoic Acid (PFHxA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorononanoic Acid (PFNA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanesulfonic Acid (PFOS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanoic Acid (PFOA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorotetradecanoic Acid (PFTeDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorotridecanoic Acid (PFTrDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroundecanoic Acid (PFUnDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoro-n-butanoic Acid (PFBA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoro-n-pentanoic Acid (PFPeA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
8:2 Fluorotelomersulfonic Acid (8:2FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
4:2 Fluorotelomersulfonic Acid (4:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoropentanesulfonic Acid (PFPeS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
6:2 Fluorotelomersulfonic Acid (6:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroheptanesulfonic Acid (PFHpS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorononanesulfonic Acid (PFNS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorodecanesulfonic Acid (PFDS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
10:2 Fluorotelomersulfonic Acid (10:2-FTS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorododecanesulfonic Acid (PFDoDS)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexadecanoic Acid (PFHxDA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctadecanoic Acid (PFODA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanesulfonamide (PFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
N-methylperfluoro-1-octanesulfonamide (NMePFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
N-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	----------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

End of DoD ELAP section of scope

Start of KY, WY, and ISO 17025 section of scope

In addition, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Other				
Perchlorate	Food & Food Products EPA 6850	EPA 6850	EPA 6850	EPA 6850
Hydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Methylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	----------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	----------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction/ Cleanup	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A
Kentucky UST Program				
Metals				
Arsenic	----------	----------	EPA 6010B	EPA 6010B
Barium	----------	----------	EPA 6010B	EPA 6010B

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Cadmium	----------	----------	EPA 6010B	EPA 6010B
Chromium	----------	----------	EPA 6010B	EPA 6010B
Lead	----------	----------	EPA 6010B	EPA 6010B
Mercury	----------	----------	EPA 7470A	EPA 7471A
Selenium	----------	----------	EPA 6010B	EPA 6010B
Silver	----------	----------	EPA 6010B	EPA 6010B

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Purgeable Organics (Volatiles)				
Diesel Range Organics (DRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Gasoline Range Organics (GRO)	----------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Wyoming Storage Tank Program				
Metals				
Cadmium	----------	----------	EPA 6010C	EPA 6010C
Chromium	----------	----------	EPA 6010C	EPA 6010C
Chromium (Total, hexavalent)	----------	----------	EPA 7196A	EPA 7196A
Lead	----------	----------	EPA 6010C	EPA 6010C
Purgeable Organics (Volatiles)				
Volatile Preparation	----------	----------	EPA 5030C EPA 5030C	EPA 5035 EPA 5035A
Benzene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
1,2-Dichloroethane	----------	----------	EPA 8260D	EPA 8260D
Diisopropyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl Benzene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl tert-butyl Ether	----------	----------	EPA 8260D	EPA 8260D
Methyl tert-butyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Naphthalene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Toluene	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Tert-amyl Methyl Ether	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Tert-butyl Alcohol	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Xylenes, total	----------	----------	EPA 5030C EPA 8260D	EPA 8260D

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Gasoline Range Organics (GRO C6-C10)	----------	----------	EPA 5030C EPA 8260D	EPA 8260D
Extractable Organics (Semivolatiles)				
Diesel Range Organics (DRO C10-C32)	----------	----------	EPA 8015C w/ EPA 3630 cleanup	EPA 8015C w/ EPA 3630 cleanup

Food and Feed (WHO 29)	**Food/Feed**			
2,3,7,8-TCDD	EPA 1613B	----------	----------	----------
2,3,7,8-TCDF	EPA 1613B	----------	----------	----------
1,2,3,7,8-PeCDF	EPA 1613B	----------	----------	----------
2,3,4,7,8-PeCDF	EPA 1613B	----------	----------	----------
1,2,3,7,8-PeCDD	EPA 1613B	----------	----------	----------
1,2,3,4,7,8-HxCDF	EPA 1613B	----------	----------	----------
1,2,3,6,7,8-HxCDF	EPA 1613B	----------	----------	----------
2,3,4,6,7,8-HxCDF	EPA 1613B	----------	----------	----------
1,2,3,7,8,9-HxCDF	EPA 1613B	----------	----------	----------
1,2,3,4,7,8-HxCDD	EPA 1613B	----------	----------	----------
1,2,3,6,7,8-HxCDD	EPA 1613B	----------	----------	----------
1,2,3,7,8,9-HxCDD	EPA 1613B	----------	----------	----------
1,2,3,4,6,7,8-HpCDF	EPA 1613B	----------	----------	----------
1,2,3,4,7,8,9-HpCDF	EPA 1613B	----------	----------	----------
1,2,3,4,6,7,8-HpCDD	EPA 1613B	----------	----------	----------
OCDF	EPA 1613B	----------	----------	----------
OCDD	EPA 1613B	----------	----------	----------
Total HpCDD	EPA 1613B	----------	----------	----------
Total HpCDF	EPA 1613B	----------	----------	----------
Total HxCDD	EPA 1613B	----------	----------	----------
Total HxCDF	EPA 1613B	----------	----------	----------
Total PeCDD	EPA 1613B	----------	----------	----------
Total PeCDF	EPA 1613B	----------	----------	----------
Total TCDD	EPA 1613B	----------	----------	----------
Total TCDF	EPA 1613B	----------	----------	----------
6 marker PCBs (PCB28, PCB52, PCB101, PCB138, PCB153, and PCB180)	EPA 1668A EPA 1668C	----------	----------	----------

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid

12 Dioxin-like PCBs (dl-PCBs)/coplanar PCBs (PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189)	EPA 1668A EPA 1668C	----------	----------	----------

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl perfluorooctane-sulfonamidoacetic acid (NetFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-methyl perfluoroctane-sulfonamidoacetic acid (NMeFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorobutanesulfonic acid (PFBS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorodecanoic acid (PFDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorododecanoic acid (PFDoDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroheptanoic acid (PFHpA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexanesulfonic acid (PFHxS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexanoic acid (PFHxA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorononanoic acid (PFNA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanesulfonic acid (PFOS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanoic acid (PFOA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorotetradecanoic acid (PFTeDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorotridecanoic acid (PFTrDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroundecanoic acid (PFUnDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoro-n-butanoic acid (PFBA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoro-n-pentanoic acid (PFPeA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
8:2 Fluorotelomersulfonic acid (8:2FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
4:2 Fluorotelomersulfonic acid (4:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoropentanesulfonic acid (PFPeS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
6:2 Fluorotelomersulfonic acid (6:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroheptanesulfonic acid (PFHpS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorononanesulfonic acid (PFNS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorodecanesulfonic acid (PFDS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
10:2 Fluorotelomersulfonic acid (10:2-FTS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorododecanesulfonic acid (PFDoDS)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexadecanoic acid (PFHxDA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctadecanoic acid (PFODA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanesulfonamide (PFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-methylperfluoro-1-octanesulfonamide (NMePFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	----------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

End of KY, WY, and ISO 17025 section of scope

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Accredited Laboratory

A2LA has accredited

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL, LLC

Lancaster, PA

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated *April 2017*).

Presented this 28th day of January 2021.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 1.01
Valid to November 30, 2022

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Katahdin Analytical Services, LLC
600 Technology Way
Scarborough, ME 04074

Fulfills the requirements of

ISO/IEC 17025:2017

and

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 01 February 2022
Certificate Number: L2223

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 AND U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V 5.3)

Katahdin Analytical Services, LLC
600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

TESTING

Valid to: **February 1, 2022** Certificate Number: **L2223**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2, 4`-DDD
GC/ECD	EPA 8081B	2, 4`-DDE
GC/ECD	EPA 8081B	2, 4`-DDT
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbon (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	CT ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 2-Trichloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloroethane
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloropropene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dichloroethane
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3-Dichloropropane
GC/MS	EPA 8260B/C; EPA 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C; EPA 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B/C; EPA 524.2	2-Butanone
GC/MS	EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C; EPA 524.2	2-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	2-Hexanone
GC/MS	EPA 8260B/C; EPA 524.2	4-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C; EPA 524.2	Acetone
GC/MS	EPA 8260B/C	Acetonitrile

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Acrolein
GC/MS	EPA 8260B/C; EPA 524.2	Acrylonitrile
GC/MS	EPA 8260B/C; EPA 524.2	Allyl chloride
GC/MS	EPA 8260B/C; EPA 524.2	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C; EPA 524.2	Bromobenzene
GC/MS	EPA 8260B/C; EPA 524.2	Bromochloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Bromodichloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Bromoform
GC/MS	EPA 8260B/C; EPA 524.2	Carbon disulfide
GC/MS	EPA 8260B/C; EPA 524.2	Carbon tetrachloride
GC/MS	EPA 8260B/C; EPA 524.2	Chlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	Chloroethane
GC/MS	EPA 8260B/C; EPA 524.2	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C; EPA 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B/C; EPA 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	Cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 8260B/C; EPA 524.2	Dibromochloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Dibromomethane
GC/MS	EPA 8260B/C; EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B/C; EPA 524.2	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C; EPA 524.2	Ethyl methacrylate
GC/MS	EPA 8260B/C; EPA 524.2	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C; EPA 524.2	Hexachlorobutadiene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C; EPA 524.2	Isopropyl benzene
GC/MS	EPA 8260B/C; EPA 524.2	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B/C; EPA 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C; EPA 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 8260B/C; EPA 524.2	Methylene chloride
GC/MS	EPA 8260B/C; EPA 524.2	Naphthalene
GC/MS	EPA 8260B/C; EPA 524.2	n-Butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	n-Propylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	o-Xylene
GC/MS	EPA 8260B/C	Pentachloroethane
GC/MS	EPA 8260B/C; EPA 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B/C; EPA 524.2	Propionitrile
GC/MS	EPA 8260B/C; EPA 524.2	sec-butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C; EPA 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Tetrahydrofuran
GC/MS	EPA 8260B/C; EPA 524.2	Toluene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B/C; EPA 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 8260B/C; EPA 524.2	Vinyl chloride
GC/MS	EPA 8260B/C	Xylene
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Disulfide
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylene chloride
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	Toluene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 8270C/D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 8270C/D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	3,4-Dimethylphenol
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	3&4-Methylphenol
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7, 12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)adipate
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 8270C/D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methy methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/D	n-Nitrosodiphenylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	O,O-Diethyl O-2pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 8270C/D	3, 3’-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3’-Dimethylbenzidine
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A/B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4, 6 -Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	4-Amino-2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene
HPLC/UV	EPA 8330A/B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/B	Tetryl
CVAA	EPA 245.1; EPA 7470A	Mercury

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7; EPA 6010C/D	Aluminum
ICP/AES	EPA 200.7; EPA 6010C/D	Antimony
ICP/AES	EPA 200.7; EPA 6010C/D	Arsenic
ICP/AES	EPA 200.7; EPA 6010C/D	Barium
ICP/AES	EPA 200.7; EPA 6010C/D	Beryllium
ICP/AES	EPA 200.7; EPA 6010C/D	Boron
ICP/AES	EPA 200.7; EPA 6010C/D	Cadmium
ICP/AES	EPA 200.7; EPA 6010C/D	Calcium
ICP/AES	EPA 200.7; EPA 6010C/D	Chromium
ICP/AES	EPA 200.7; EPA 6010C/D	Cobalt
ICP/AES	EPA 200.7; EPA 6010C/D	Copper
ICP/AES	EPA 200.7; EPA 6010C/D	Iron
ICP/AES	EPA 200.7; EPA 6010C/D	Lead
ICP/AES	EPA 200.7; EPA 6010C/D	Magnesium
ICP/AES	EPA 200.7; EPA 6010C/D	Manganese
ICP/AES	EPA 200.7; EPA 6010C/D	Molybdenum
ICP/AES	EPA 200.7; EPA 6010C/D	Nickel
ICP/AES	EPA 200.7; EPA 6010C/D	Potassium
ICP/AES	EPA 200.7; EPA 6010C/D	Selenium
ICP/AES	EPA 200.7; EPA 6010C/D	Silicon
ICP/AES	EPA 200.7; EPA 6010C/D	Silver
ICP/AES	EPA 200.7; EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Strontium
ICP/AES	EPA 200.7; EPA 6010C/D	Thallium
ICP/AES	EPA 200.7; EPA 6010C/D	Tin
ICP/AES	EPA 200.7; EPA 6010C/D	Titanium
ICP/AES	EPA 200.7; EPA 6010C/D	Vanadium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/AES	EPA 200.7; EPA 6010C/D	Zinc
ICP/MS	EPA 200.8; EPA 6020A/B	Aluminum
ICP/MS	EPA 200.8; EPA 6020A/B	Antimony
ICP/MS	EPA 200.8; EPA 6020A/B	Arsenic
ICP/MS	EPA 200.8; EPA 6020A/B	Barium
ICP/MS	EPA 200.8; EPA 6020A/B	Beryllium
ICP/MS	EPA 200.8; EPA 6020A/B	Boron
ICP/MS	EPA 200.8; EPA 6020A/B	Cadmium
ICP/MS	EPA 200.8; EPA 6020A/B	Calcium
ICP/MS	EPA 200.8; EPA 6020A/B	Chromium
ICP/MS	EPA 200.8; EPA 6020A/B	Cobalt
ICP/MS	EPA 200.8; EPA 6020A/B	Copper
ICP/MS	EPA 200.8; EPA 6020A/B	Iron
ICP/MS	EPA 200.8; EPA 6020A/B	Lead
ICP/MS	EPA 200.8; EPA 6020A/B	Magnesium
ICP/MS	EPA 200.8; EPA 6020A/B	Manganese
ICP/MS	EPA 200.8; EPA 6020A/B	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A/B	Nickel
ICP/MS	EPA 200.8; EPA 6020A/B	Potassium
ICP/MS	EPA 200.8; EPA 6020A/B	Selenium
ICP/MS	EPA 200.8; EPA 6020A/B	Silver
ICP/MS	EPA 200.8; EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 200.8; EPA 6020A/B	Thallium
ICP/MS	EPA 200.8; EPA 6020A/B	Tin
ICP/MS	EPA 200.8; EPA 6020A/B	Tungsten
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8; EPA 6020A/B	Vanadium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A/B	Zinc
IC	EPA 300.0; EPA 9056A	Chloride
IC	EPA 300.0; EPA 9056A	Fluoride
IC	EPA 300.0; EPA 9056A	Nitrate as N
IC	EPA 300.0; EPA 9056A	Nitrite as N
IC	EPA 300.0; EPA 9056A	Nitrate + Nitrite
IC	EPA 300.0; EPA 9056A	Sulfate
IC	SOP CA-776	Lactic Acid
IC	SOP CA-776	Acetic Acid
IC	SOP CA-776	Propionic Acid
IC	SOP CA-776	Formic Acid
IC	SOP CA-776	Butyric Acid
IC	SOP CA-776	Pyruvic Acid
IC	SOP CA-776	i-Pentanoic Acid
IC	SOP CA-776	Pentanoic Acid
IC	SOP CA-776	i-Hexanoic Acid
IC	SOP CA-776	Hexanoic Acid
Titration	EPA 310.1; SM 2320B	Alkalinity
Caculation	SM 2340B	Hardness
Gravimetric	EPA 1664A; EPA 9070A	Oil and Grease, Oil and Grease with SGT
Gravimetric	SM 2540B/C/D	Solids
ISE	EPA 120.1; SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH

Non-Potable Water		
Technology	**Method**	**Analyte**
Titration	SM 2340C	Hardness
Titration	SM 4500SO_3 B	Sulfite
Titration	EPA 9034; SM 4500-S^{2-} F	Sulfide
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
IR	EPA 9060A; SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1; SM 2130B	Turbidity
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 335.4; EPA 9012B; SM 4500-CN G	Amenable cyanide
UV/VIS	EPA 350.1; SM 4500-NH3 H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrate + Nitrite
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrate as N
UV/VIS	EPA 353.2; SM 4500-NO3 F	Nitrite as N
UV/VIS	EPA 365.2; SM 4500-P E	Orthophosphate as P
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1; EPA 9065	Total Phenolics
UV/VIS	SM 4500-Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS
UV/VIS	EPA 7196A; SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B; EPA 335.4	Total Cyanide
UV/VIS	EPA 9251; SM 4500-Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide

Non-Potable Water		
Preparation	**Method**	**Type**
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2,4`-DDD
GC/ECD	EPA 8081B	2,4`-DDE
GC/ECD	EPA 8081B	2,4`-DDT
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	Cis-Nonachlor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	Trans-Nonachlor
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbons (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	MA DEP EPH EPA 3546	Extractable Petroleum Hydrocarbons Microwave Extraction Preparation
GC/FID	CT-ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B/C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1, 1, 2-Trichloroethane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	1, 1-Dichloroethane
GC/MS	EPA 8260B/C	1, 1-Dichloroethylene
GC/MS	EPA 8260B/C	1, 1-Dichloropropene
GC/MS	EPA 8260B/C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 2-Dibromoethane
GC/MS	EPA 8260B/C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 2-Dichloroethane
GC/MS	EPA 8260B/C	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 3-Dichloropropane
GC/MS	EPA 8260B/C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	2, 2-Dichloropropane
GC/MS	EPA 8260B/C	2-Butanone
GC/MS	EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C	2-Chlorotoluene
GC/MS	EPA 8260B/C	2-Hexanone
GC/MS	EPA 8260B/C	4-Chlorotoluene
GC/MS	EPA 8260B/C	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C	Acetone
GC/MS	EPA 8260B/C	Acetonitrile

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Acrolein
GC/MS	EPA 8260B/C	Acrylonitrile
GC/MS	EPA 8260B/C	Allyl chloride
GC/MS	EPA 8260B/C	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C	Bromobenzene
GC/MS	EPA 8260B/C	Bromochloromethane
GC/MS	EPA 8260B/C	Bromodichloromethane
GC/MS	EPA 8260B/C	Bromoform
GC/MS	EPA 8260B/C	Carbon disulfide
GC/MS	EPA 8260B/C	Carbon tetrachloride
GC/MS	EPA 8260B/C	Chlorobenzene
GC/MS	EPA 8260B/C	Chloroethane
GC/MS	EPA 8260B/C	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B/C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	cis-1,3-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 8260B/C	Dibromochloromethane
GC/MS	EPA 8260B/C	Dibromomethane
GC/MS	EPA 8260B/C	Dichlorodifluoromethane
GC/MS	EPA 8260B/C	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B/C	Ethyl methacrylate
GC/MS	EPA 8260B/C	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C	Isopropyl benzene
GC/MS	EPA 8260B/C	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B/C	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 8260B/C	Methylene chloride
GC/MS	EPA 8260B/C	Naphthalene
GC/MS	EPA 8260B/C	n-Butylbenzene
GC/MS	EPA 8260B/C	n-proplybenzene
GC/MS	EPA 8260B/C	o-Xylene
GC/MS	EPA 8260B/C	pentachloroethane
GC/MS	EPA 8260B/C	p-Isopropyltoluene
GC/MS	EPA 8260B/C	Propionitrile
GC/MS	EPA 8260B/C	sec-butylbenzene
GC/MS	EPA 8260B/C	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 8260B/C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B/C	Tetrahydrofuran

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Toluene
GC/MS	EPA 8260B/C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B/C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C	Trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B/C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 8260B/C	Vinyl chloride
GC/MS	EPA 8260B/C	Xylene
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dichlorophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 8270C/D	2, 4-Dinitrotoluene (2 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 8270C/D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3, 3`-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C/D	3, 4-Dimethylphenol
GC/MS	EPA 8270C/D	3&4-Methylphenol
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	4-Nitroaniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270C/D	Bis(2-Ethylhexyl)adipate
GC/MS	EPA 8270C/D	Butyl benzyl phthalate

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methyl methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O, O, O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	O,O-Diethyl O-2-pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	3,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A	1 ,3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4, 6 –Dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6,Dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene
HPLC/UV	EPA 8330B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330B	Tetryl
HPLC/UV	EPA 8330B	Nitroguanidine
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010C/D	Aluminum
ICP/AES	EPA 6010C/D	Antimony
ICP/AES	EPA 6010C/D	Arsenic
ICP/AES	EPA 6010C/D	Barium
ICP/AES	EPA 6010C/D	Beryllium
ICP/AES	EPA 6010C/D	Boron
ICP/AES	EPA 6010C/D	Cadmium
ICP/AES	EPA 6010C/D	Calcium
ICP/AES	EPA 6010C/D	Chromium
ICP/AES	EPA 6010C/D	Cobalt
ICP/AES	EPA 6010C/D	Copper
ICP/AES	EPA 6010C/D	Iron
ICP/AES	EPA 6010C/D	Lead
ICP/AES	EPA 6010C/D	Magnesium
ICP/AES	EPA 6010C/D	Manganese
ICP/AES	EPA 6010C/D	Molybdenum
ICP/AES	EPA 6010C/D	Nickel
ICP/AES	EPA 6010C/D	Potassium
ICP/AES	EPA 6010C/D	Selenium
ICP/AES	EPA 6010C/D	Silicon
ICP/AES	EPA 6010C/D	Silver
ICP/AES	EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Strontium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010C/D	Thallium
ICP/AES	EPA 6010C/D	Tin
ICP/AES	EPA 6010C/D	Titanium
ICP/AES	EPA 6010C/D	Vanadium
ICP/AES	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Boron
ICP/MS	EPA 6020A/B	Cadmium
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 6020A/B	Thallium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Tungsten
ICP/MS	EPA 6020A/B	Vanadium
ICP/MS	EPA 6020A/B	Zinc
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Orthophosphate
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9071A/B	Oil and Grease, Oil and Grease with SGT
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
Titration	Walkley-Black	Total Organic Carbon
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 350.1; SM 4500-NH3 H	Ammonia as N
UV/VIS	EPA 9251; SM 4500-Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	SM 3500-Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Sieves, Hydrometer	ASTM D422	Grain Size

Solid and Chemical Materials		
Preparation	**Method**	**Type**
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up
Cleanup Methods	EPA 3620C	Florsil Clean-up
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction
Organic Preparation	EPA 3546	Microwave Extraction Preparation for EPA 8082A, 8081B and 8270C, D, 8015C/D
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap
Organic Preparation	EPA 8330A/B	ISM

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	1, 1, 1-Trichloroethane
GC/MS	EPA TO-15	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA TO-15	1, 1, 2-Trichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethylene
GC/MS	EPA TO-15	1, 2, 4-Trichlorobenzene
GC/MS	EPA TO-15	1, 2, 4-Trimethylbenzene
GC/MS	EPA TO-15	1, 2-Dibromoethane (EDB)
GC/MS	EPA TO-15	1, 2-Dichlorobenzene

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	1, 2-Dichloroethane
GC/MS	EPA TO-15	1, 2-Dichloroethenes (Total)
GC/MS	EPA TO-15	1, 2-Dichloropropane
GC/MS	EPA TO-15	1, 3, 5-Trimethylbenzene
GC/MS	EPA TO-15	1, 3-Butadiene
GC/MS	EPA TO-15	1, 3-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dioxane
GC/MS	EPA TO-15	1,1,2-Trichloro1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
GC/MS	EPA TO-15	1,4-Difluorobenzene
GC/MS	EPA TO-15	2,2,4-Trimethylpentane
GC/MS	EPA TO-15	2-Butanone
GC/MS	EPA TO-15	2-Chlorotoluene
GC/MS	EPA TO-15	2-Hexanone
GC/MS	EPA TO-15	2-Propanol
GC/MS	EPA TO-15	3-Chloropropene (Allyl chloride)
GC/MS	EPA TO-15	4-Ethyltoluene
GC/MS	EPA TO-15	4-Methyl-2-pentanone
GC/MS	EPA TO-15	Acetone
GC/MS	EPA TO-15	Acrolein
GC/MS	EPA TO-15	Benzene
GC/MS	EPA TO-15	Benzyl chloride
GC/MS	EPA TO-15	Bromochloromethane
GC/MS	EPA TO-15	Bromodichloromethane
GC/MS	EPA TO-15	Bromoform
GC/MS	EPA TO-15	Carbon disulfide
GC/MS	EPA TO-15	Carbon tetrachloride

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Chlorobenzene
GC/MS	EPA TO-15	Chloroethane
GC/MS	EPA TO-15	Chloroform
GC/MS	EPA TO-15	Cis-1, 2-Dichloroethene
GC/MS	EPA TO-15	Cis-1, 3-Dichloropropene
GC/MS	EPA TO-15	Cyclohexane
GC/MS	EPA TO-15	Dibromochloromethane
GC/MS	EPA TO-15	Dichlorodifluoromethane (Freon 12)
GC/MS	EPA TO-15	Ethanol
GC/MS	EPA TO-15	Ethyl acetate
GC/MS	EPA TO-15	Ethylbenzene
GC/MS	EPA TO-15	Hexachlorobutadiene
GC/MS	EPA TO-15	Isopropyl alcohol
GC/MS	EPA TO-15	Isopropylbenzene
GC/MS	EPA TO-15	m, p-Xylene
GC/MS	EPA TO-15	Methyl bromide (Bromomethane)
GC/MS	EPA TO-15	Methyl chloride (Chloromethane)
GC/MS	EPA TO-15	Methyl methacrylate
GC/MS	EPA TO-15	Methyl tert-butyl ether
GC/MS	EPA TO-15	Methylene chloride
GC/MS	EPA TO-15	Naphthalene
GC/MS	EPA TO-15	n-Heptane
GC/MS	EPA TO-15	n-Hexane
GC/MS	EPA TO-15	o-Xylene
GC/MS	EPA TO-15	Propene
GC/MS	EPA TO-15	Styrene
GC/MS	EPA TO-15	tert-Butyl alcohol
GC/MS	EPA TO-15	Tetrachloroethylene (Perchloroethylene)

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Tetrahydrofuran
GC/MS	EPA TO-15	Toluene
GC/MS	EPA TO-15	trans-1, 2-Dichloroethylene
GC/MS	EPA TO-15	trans-1, 3-Dichloropropylene
GC/MS	EPA TO-15	Trichloroethene (Trichloroethylene)
GC/MS	EPA TO-15	Trichlorofluoromethane (Freon 11)
GC/MS	EPA TO-15	Vinyl acetate
GC/MS	EPA TO-15	Vinyl bromide
GC/MS	EPA TO-15	Vinyl chloride
GC/MS	EPA TO-15	Xylenes (Total)
GC/MS	MA DEP APH	Aliphatic C5-C8 range
GC/MS	MA DEP APH	Aliphatic C9-C12 range
GC/MS	MA DEP APH	Aromatic C9-C10 range
GC/MS	MA DEP APH	1,3-Butadiene
GC/MS	MA DEP APH	Benzene
GC/MS	MA DEP APH	Ethylbenzene
GC/MS	MA DEP APH	m+p-Xylene
GC/MS	MA DEP APH	Methyl tert-butyl ether
GC/MS	MA DEP APH	Naphthalene
GC/MS	MA DEP APH	o-Xylene
GC/MS	MA DEP APH	Toluene

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane/cis-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane/trans-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/D SIM	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D SIM	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
ICP/AES	EPA 6010C/D	Aluminum
ICP/AES	EPA 6010C/D	Antimony
ICP/AES	EPA 6010C/D	Arsenic
ICP/AES	EPA 6010C/D	Barium

Biological Tissue		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010C/D	Beryllium
ICP/AES	EPA 6010C/D	Boron
ICP/AES	EPA 6010C/D	Cadmium
ICP/AES	EPA 6010C/D	Calcium
ICP/AES	EPA 6010C/D	Chromium
ICP/AES	EPA 6010C/D	Cobalt
ICP/AES	EPA 6010C/D	Copper
ICP/AES	EPA 6010C/D	Iron
ICP/AES	EPA 6010C/D	Lead
ICP/AES	EPA 6010C/D	Magnesium
ICP/AES	EPA 6010C/D	Manganese
ICP/AES	EPA 6010C/D	Molybdenum
ICP/AES	EPA 6010C/D	Nickel
ICP/AES	EPA 6010C/D	Potassium
ICP/AES	EPA 6010C/D	Selenium
ICP/AES	EPA 6010C/D	Silver
ICP/AES	EPA 6010C/D	Sodium
ICP/AES	EPA 6010C/D	Thallium
ICP/AES	EPA 6010C/D	Tin
ICP/AES	EPA 6010C/D	Vanadium
ICP/AES	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Boron
ICP/MS	EPA 6020A/B	Cadmium

Biological Tissue		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Thallium
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Vanadium
ICP/MS	EPA 6020A/B	Zinc
CVAA	EPA 7471B	Mercury

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2223.

R. Douglas Leonard Jr., VP, PILR SBU

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UFP-QAPP Addendum Appendix D

Field Standard Operating Procedures

- EA-SOP-007 Surface Water Sampling
- EA-SOP-043 Multi-Probe Water Quality Monitoring Instruments
- EA-SOP-057 Incremental Sampling Methodology

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Standard Operating Procedure No. 007
for
Surface Water Sampling

Prepared by

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Revision: 2
March 2020

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PROJECT-SPECIFIC VARIANCE FORM

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☐ **B. No variances**

SOP No. 007	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	March 2020	Systematic Review and Update	Jason Stroup, Kevin Kowalk	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for sampling surface water. This procedure can be applied to the collection of surface water samples from marine and estuarine systems, streams, rivers, ditches, lakes, ponds, and lagoons. Surface water samples provide an indication of the amount of contaminant in the surface water. It is, therefore, important to collect a representative sample.

2. MATERIALS

The following materials may be required:

0.45-micrometer (µm) disposable filters	Sample bottles
Cooler with ice	Short-handled dip sampler (PTFE or stainless steel)
Long-handled dip sampler (polytetrafluoroethelyne [PTFE] or stainless steel)	Stainless steel or PTFE-lined bucket
Peristaltic pump	Niskin bottle (or similar sampling device)
Disposable peristaltic head tubing	Disposable Teflon and/or Teflon lined poly tubing

3. PROCEDURE

For all surface water samples, use a Global Positioning System to record sampling coordinates and mark the sampling locations on a site map. Photograph (if cameras are allowed onsite) and describe each location, place a numbered stake above the visible high-water mark on the bank closest to the sampling location, and/or mark adjacent trees with surveyor's flagging. The photographs and descriptions must be adequate to allow the sampling station to be relocated at some future date by someone other than the original sampling crew. Use a long-handled dip sampler where access is poor or non-contact with water is suggested in the Health and Safety Plan.

Sampling should be performed deliberately and methodically to minimize disturbance of bottom sediments, yet as quickly as possible to ensure a representative sample. If wading in a stream, sample upstream at the sampling location to prevent disturbance of the stream bottom from impacting the sample. To prevent contamination of the exterior of the sample container, and/or potential contamination of the surface water sample by laboratory contaminants on the exterior of the bottle, the sample container should never be dipped into the water; rather, a decontaminated sampling device should be used to collect unfiltered samples.

Sampling with the PTFE or stainless steel sampler (long-handled or measuring cup-type):

- Remove the cap from the sample bottle.
- Dip a sample of surface water using the sampler.

- Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.
- Add preservative as required. Replace cap, and place in cooler immediately.

Sampling with stainless steel or PTFE-lined bucket:

- Remove cap from sample bottle.
- Gently dip collection bucket in the water. Fill bucket and carefully lift from water body.
- Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.
- Add preservative as required. Replace cap, and place in cooler immediately.

– OR –

- Use smaller sampling cup to transfer sample from bucket to sample bottle as described above.

Sampling with a Niskin bottle (or similar device):

- Prepare the bottle for deployment by placing the ends of the bottle in the open position and lock the ends into the trigger mechanism.
- Lower the bottle to the desired depth of sampling (on either a wire cable or rope).
- Place a messenger (triggering device) on the cable/rope and deploy by allowing free-fall down the cable/rope.
- Bring the bottle back to the surface and pour sample into a sample container.

Sampling with a peristaltic pump and Teflon® or Teflon® lined tubing:

- Cut a length of Teflon® tubing to the depth of sampling specified by the client or project-specific Sampling and Analysis Plan.
- Insert one end of the tubing into the intake hose on the peristaltic pump.
- Place a stainless steel weight on the tubing and lower to the specified depth;
- Cut a length of tubing and insert into the output (out-flow) hose on the peristaltic pump.

- After applying power to the peristaltic pump, proceed to pump site water through the tubing apparatus. The hose volume should be pumped approximately five times through the tubing before sampling.

- Fill the required sample containers.

- If filtering is required, obtain filtered sample by placing a 0.45-µm in-line filter on the end of the output tube and fill the required sample containers.

Both filtered and unfiltered samples may be required for metals analyses. Bulk samples for filtration will be collected using the stainless steel or PTFE-lined bucket method described above. Sample filtration must be performed immediately upon retrieval of the bulk sample as follows.

Filtration will be performed immediately after collecting sample. Set up filtration equipment prior to collecting sample. Filtration may be accomplished by gravity or, if necessary due to slow filtering, a peristaltic pump will be used to pressure filter the sample. Vacuum filtration will not be used due to the possibility of analyte volatilization.

Gravity filtration will be accomplished as follows:

- Using decontaminated forceps, place a 0.45-µm membrane in a decontaminated filter funnel.

- Slowly pour sample into the funnel and collect filtrate directly into appropriate sample container(s).

- Add preservative(s) as required by project-specific Sampling and Analysis Plan. Immediately cap container and place in cooler.

- Dispose of filter membrane.

Pressure filtration will be accomplished as follows:

- Using previously assembled disposable tubing, 45-µm in-line filter, and peristaltic pump, filter sample from collection bucket into appropriate container.

- Adjust pump rate to avoid aeration of sample.

- Fill container, add preservative as required immediately cap container, and place in cooler.

- Dispose of filter and tubing.

4. MAINTENANCE

Refer to manufacturer's specifications for maintenance procedures on generators and pumps.

5. PRECAUTIONS

The following precautions should be taken:

- Avoid disturbing bottom sediments.
- Consult the Health and Safety Plan prior to collecting any samples for personal protective equipment such as dermal and respiratory protection and personal flotation devices when sampling in or near deep water or from boats.
- Always decontaminate the sampling and filtration equipment, and change gloves between sampling locations to minimize the risk of cross-contamination.

6. REFERENCES

None.

Standard Operating Procedure No. 043 for Multi-Probe Water Quality Monitoring Instruments

Prepared by

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Revision No. 2
June 2020

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PROJECT-SPECIFIC VARIANCE FORM

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☐ **B. No variances**

SOP No. 043	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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ATTACHMENT A: EQUIPMENT CALIBRATION LOG

DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
2	June 2020	Systematic review and update. Combined with previously separate SOPs for pH, temperature, specific conductivity, turbidity, dissolved oxygen, and redox potential.	Eddie Meadows Catherine Maxwell	Matthew Bowman

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for field operation of multi-probe water quality instruments. The instrument can monitor a variety of basic parameters including dissolved oxygen, percent saturation, temperature, pH, conductivity, specific conductivity, resistivity, salinity, total dissolved solids, turbidity, oxidation reduction potential (ORP), level, and depth.

The use of brand names in this SOP is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the vendor must provide applicable and comparable SOPs for the maintenance and calibration from the specific manufacturer of the instrument being used.

2. MATERIALS

The following materials may be required:

- Multi-probe instrument
- Probe/sonde with appropriate cables
- Appropriate standards/calibration fluids
- Accessories (batteries, charger, case, etc.)
- Decontamination materials or laboratory wipes
- Deionized water and distilled water (as needed for calibration and decontamination)
- Instrument logbook
- Manufacturer's Operations Manual.

3. CALIBRATION PROCEDURE

Calibration must be performed or verified daily at a minimum before using the instrument. Calibration may be performed in the laboratory or in the field. Detailed step-by-step calibration procedures for the equipment described below are provided in the most recent version of the manufacturer's Operations Manual. Documentation includes at a minimum: time, date, analyst, standard, primary standard/calibration fluid lot number, secondary standard/calibration fluid lot number, and expiration dates of standards/calibration fluids. An example calibration log is provided in Attachment A.

Fill the calibration cup with the appropriate standard as follows:

- Temperature: None required
- Specific Conductance: Conductivity standards
- pH: pH 7 buffer plus pH 4 and/or pH 10 buffer
- Dissolved Oxygen: Saturated air or saturated water
- ORP: Quinhydrone (Zobell's Solution) or other standard

- Turbidity: Nephelometric turbidity unit (NTU) standards
- Salinity: Calibration for specific conductance
- Depth/Level: Set zero in air.

3.1 CONDUCTIVITY CALIBRATION

Conductivity meters are calibrated at least once per day to at least one standard. The standard should be selected in accordance with the range expected to be measured (e.g., 1.0 microSiemans per centimeter [µS/cm]) standard should not be used to calibrate meters being used in saltwater). See manufacturer's recommendations in the Operations Manual for additional information on calibration standard selection. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.2 PH CALIBRATION

The pH meters are calibrated at least once per day to a minimum of two standard buffers (pH 4 and 7, or pH 7 and 10) in accordance with the range expected to be measured. The calibration is verified using a fresh solution of pH 7 buffer post-calibration. The probe should be rinsed in distilled water between standards. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.3 DISSOLVED OXYGEN CALIBRATION

Dissolved oxygen meters are saturated-air or saturated-water calibrated at least once per day. Each method requires the true barometric pressure to be input or collected from the instrument prior to calibration.

- ***Saturated Air Method***—Dip the calibration chamber (i.e., probe storage cup) into distilled or tap water at ambient temperature, pour out excess water, and then insert dissolved oxygen probe into the wet chamber. This ensures that the air inside the chamber is saturated with water vapor. CAUTION: Be sure that the membrane/probe has no droplets of water adhering to it since this would reduce the rate of oxygen diffusion through the membrane and would produce erroneous results. Do not fully thread the probe storage cup on the probe during equilibration.

- ***Saturated Water Method***—To make a 100 percent (%) air-saturated calibration standard, fill a container (e.g., a 1-liter or 1-gallon container with a closed top) three-quarters full with distilled water or clean (conductivity of less than 500 µS/cm) tap water. Let the water temperature reach equilibrium with the calibration environment. Then shake the container vigorously for approximately 30 seconds. This makes 100% air-saturated water. Place the air-saturated water into the probe storage cup and allow to equilibrate. Do not fully thread the probe storage cup on the probe during equilibration.

Calibration information is recorded in conjunction with the data collected for that sampling event.

3.4 OXIDATION REDUCTION POTENTIAL CALIBRATION

ORP meters are calibrated at least once per day to at least one standard. It is recommended that Zobell's Solution is used; however, another solution can be used as long as it meets the manufacturer's specifications for calibration. Note that the standard value for Zobell's Solution is dependent on temperature. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.5 TURBIDITY CALIBRATION

The turbidity meters are calibrated at least once per day to a minimum of two standards (0 NTU and 100 or 200 NTUs recommended) in accordance with the range expected to be measured. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.6 DEPTH/LEVEL CALIBRATION

The depth and level calibration is performed with the depth sensor module in the air and not immersed in any solution. The appropriate correction for height above the water surface is inputted into the meter. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.7 ADDITIONAL CALIBRATIONS

Additional measurements may be taken with the multi-probe water quality instruments. For any of these measurements, the calibration procedures will be conducted in accordance with the manufacturer's specifications. Calibration information is recorded in conjunction with the data collected for that sampling event.

4. FIELD OPERATION

4.1 SETUP OF MULTI-PROBE WATER QUALITY INSTRUMENT

Post-calibration and prior to sampling, the multi-probe water quality instrument should be inspected, cleaned, and set up for data collection. If the cables have been unattached, they will be reconnected to the transmitter (if applicable) and the display. Once all cables are attached, the meter will be turned on and allowed to warm up for a few seconds in order to allow the display screen to load. The unit should be allowed to come to ambient air temperature if it has been stored in a hotter or colder environment prior to use.

4.2 SURFACE WATER

Prior to sampling, check the condition of the probes before each deployment. When sampling in surface water, the sensor must be in an amount of water sufficient for all probes to be submerged. Data values displayed on the display screen are recorded in a field logbook, a dedicated project field form (i.e., an EA Purging and Sampling Record, or on an EA-provided iPad on an approved GoFormz), and accepted into the instrument's data logger (if used). Post-data collection, the sensor will be retrieved and rinsed for use at the next sample location. If travel time between sample locations is significant, the display is to be turned off. When all sampling is completed, disconnect all equipment, clean probes and the instrument in accordance with the manufacturer's instructions, attach a solid protective cap, and return it to its proper storage location.

4.3 GROUNDWATER

Prior to sampling, check the condition of the probes before each deployment. When sampling groundwater, mount sampler on a flow-through cell. Start sampler pump and allow pump/hose system to be purged of air bubbles. Required parameters should be recorded every 3-5 minutes (unless otherwise specified in the sampling plan). Record the monitored values in the appropriate field logbook, on a dedicated project field form (i.e., an EA Purging and Sampling Record, or on an EA provided iPad on an approved GoFormz) to ensure against inadvertent data loss. If travel time between sample locations is significant, the display is to be turned off. When all sampling is completed, disconnect all equipment, clean probes and the instrument in accordance with the manufacturer's instruction, remove flow-through cell and attach solid protective cap, and return it to its proper storage location. If a flow-through cell cannot be used (e.g., groundwater sampling using a bailer), bailed water should be poured into a clean container for collecting readings over standard intervals of volume purged or time.

5. MAINTENANCE

All maintenance should be performed in accordance with the manufacturer's Operations Manual.

6. PRECAUTIONS

Check the condition of the probes frequently between sampling. Do not force pins into connections; note keying sequence. If field readings are outside the expected range, check for bubbles on, or damage to, the probes. If there are no bubbles or damage, recalibrate the sensor.

7. REFERENCES

Not applicable.

Attachment A

Equipment Calibration Log

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Standard Operating Procedure No. 057
for
Incremental Sampling Methodology

Prepared by

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Revision 1
March 2020

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☐ **B. No variances**

SOP No. 057	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	March 2020	Update with new applications/guidance	Daniel Hinckley, Brenda Nuding	Matthew Bowman

1. INTRODUCTION

1.1 SCOPE AND APPLICATION

The incremental sampling methodology (ISM) technique, also known as the multi-incremental (MI) sampling approach, offers reduced sample variability and improved sample representativeness for surface and subsurface soils as well as sediments than traditional discrete or composite sampling approaches. This standard operating procedure (SOP) presents a brief summary of the background of the ISM as well as scope, application, general concepts, equipment and materials, procedures to be followed for implementation, and precautions. The purpose of this SOP is to delineate protocols for the application of ISM field sampling of surface soil. The procedure, which can be adapted to allow for MI sampling in other environments (i.e., in an excavation trench or spoil piles), has been adapted from U.S. Army Corps of Engineers (2009) and Interstate Technology and Regulatory Council (ITRC) 2012 sampling guidance.

ITRC has published a comprehensive guidance for ISM (ITRC 2012). This document presents details about the statistical theories on which it is based, how to establish and sample decision units (DUs), field and laboratory processing and subsampling of ISM samples, and other related topics. U.S. Environmental Protection Agency's sample design software Visual Sampling Plan, available at https://vsp.pnnl.gov/ at no cost, includes a module that will model the appropriate number of increments (aliquots) to be collected within each DU and will design a sample plan that will achieve the data quality objectives of a project.

The suitability of ISM should be evaluated on a case-by case basis when planning documents are prepared for an environmental investigation to ensure that the resulting analytical data are appropriate to make the decisions required by the project. This evaluation process considers:

1. Planning elements based on the decisions to be made for each potentially complete pathway (based on the conceptual site model), including contaminant distribution, potential hotspot size, future land use scenarios, contaminant fate and transport, regulatory acceptance, etc.

2. Sample preparation procedures to be employed by the analytical laboratory (limitations and impacts on the analytical data due to the various preparation methods that can be employed and the nature of the contaminant to be analyzed).

3. Data evaluation requirements (i.e., the data needing to meet a certain level of confidence). In addition to technical considerations, stakeholders' input should be folded into the planning stages. Consequently, specific field requirements may be outlined in the planning documents for the sampling program implementation to supplement this SOP.

This SOP focuses on the actual collection of ISM samples, not project planning or data evaluation to follow, and assumes that successful project planning and scoping have been performed, documented, and agreed to by stakeholders. Because DUs are defined so that the

exposure point concentration value (estimate of the mean) obtained is relevant to an explicitly articulated end use of the data, it is imperative that **changes to the DUs or sampling strategy deemed necessary by actual field conditions unanticipated at the time the sampling plan was designed should be made by the project technical lead rather than by field personnel.** This way, field deviations from the approved plan during sample collection will not negatively impact the adequacy of the data for the planned purpose.

1.2 GLOSSARY OF TERMS

1.2.1 Decision Units

A DU is a specific area (or volume of soil) about which a decision is to be made. In the ideal and most direct case, the DU and sample unit (SU) are the same; however, a DU may be composed of a single SU, or may include multiple SUs. It is important that the contamination distribution and future use/exposure scenario for the entire area of a DU or SU are consistent. Either the entire or a portion of the SUs composing the DU may be sampled using ISM, and the number of SUs sampled depends on the confidence of the data that are extended from the SUs to the DU. ITRC (2012) provides a Microsoft Excel spreadsheet that allows for the calculation of spatially-weighted DU exposure concentrations from SU exposure concentrations.

1.2.2 Grid Cell

A grid cell is a subdivision of the DU. DUs may be divided into uniform-size grid cells, and one increment is collected from each cell, from the same (or different depending on the sample design) relative location within each grid cell. The shape of the cells is not specified—the only criterion for cell shape selection is that the cells should be of equal size (they can be triangular, square, rectangular, etc.) so the increments collected from each cell are equally weighted over the DU.

1.3 GENERAL CONCEPTS

The use of standard discrete samples to characterize soil contamination has two significant sources of error:

1. Field sampling error is at least 10 times greater than analytical (laboratory-associated) error.

2. A source of analytical error was found to be that in sample processing and subsampling (a single subsample from the 4- or 8-ounce soil jar is taken at the laboratory).

Depending on the areal and vertical contaminant distribution profile, ISM sampling and processing are designed to minimize these sources of error, resulting in an average concentration that is a more precise and accurate estimate of the average contaminant concentration for the DU.

It is also important to note that the horizon characterized by ISM sampling is usually superficial, although ISM may be implemented at greater depth, resulting in much higher associated sampling costs.

2. EQUIPMENT AND MATERIALS

The following equipment and materials may be required:

- Spray paint[1], pin flags, or rope to mark either grid corners or outline the sampling grid
- Incremental sampling tool (i.e., the MI tool developed by the Cold Regions Research and Engineering Laboratory or alternative[2] coring device); stainless-steel spoons or scoops may be used, such that aliquots of equal mass or volume are collected from each aliquot location
- Clean resealable (i.e., Zip-lock®) bags, 5-gallon plastic containers, or other appropriate large container for placing the increments; the size of the container should be adequate to hold the entire ISM sample volume, which is approximately 1-2 kilograms
- Coolers and ice for cold storage of samples after collection
- Field logbook and pen with waterproof black ink for field documentation
- Global Positioning System (GPS) instrument or other survey equipment to document locations of DU or SUs
- Personal protective equipment to be worn during sample collection as required by the Health and Safety Plan for the project.

If ISM sampling is used for volatile organic compound analysis, the increments of equal mass are collected with tools such as EnCore® sampler and placed in a pre-tared container obtained from the analytical laboratory partially filled with methanol. Note the limitations for sample shipment presented in Section 5, Precautions. Multiple methanol preserved sample containers may be used for one ISM sample if necessary.

3. INCREMENTAL SAMPLING METHODOLOGY PROCEDURE

Increments (typically 30 and up to 50 based on project-specific sampling design) of soil of approximately of the same weight or volume will be collected within each cell of the DU.

[1] Avoid if spray paint is likely to affect MI sample quality.
[2] A source for the MI sampling tool shown in this SOP is Ike Loukos, LES Engineering, Inc. Telephone No. 301-471-3393, email i.loukos@att.net.

For surface soil sampling, a coring tool may be used to facilitate the rapid collection of uniform, representative increments from a consistent depth interval. This way, equal volumes are collected for each increment and equal mass is obtained under the assumption that the density of the sampled medium is uniform across the cell of the DU. The size of the coring tool will be selected based on the volume of the increments, which is in turn calculated based on number and depth of the increments and the fact that an adequate total sample mass is typically 1-2 kilograms dry weight (to overcome effects of compositional heterogeneity due to the inherent particulate nature of soil and sediment). It is not necessary to determine the location of every increment collected using a GPS instrument, as long as the areal extent of the DU or SU within a DU has been properly identified.

The following procedure will be followed within each DU.

- Demarcate the areal extent of the DU in the field using pin flags, spray paint, or rope and fixed with a GPS instrument. Select increments as defined in the project-specific sampling plan.

- Prior to ISM sampling activities, don the personal protective equipment. Then, collect increments from the depth interval specified in the planning documents (usually up to 6 inches deep) using a coring tool or other method that ensures equal volume is collected for each increment. Unless specifically excluded in the sampling plan, include the vegetative mat in the sampled interval. Note that some plans may require sampling of native soil only; the horizontal limits of sampling will be dependent on past disposal practices and the decision to be made. If used, push the stainless-steel sampler into the soil until the sampler is full and will not penetrate further. Remove the sampler carefully, and push the soil out of the sampler with the lever on the side of the instrument (see photos below).

- Place the sample (increment or aliquot) directly into a large re-sealable bag, 5-gallon bucket, or alternative container (note the above photos show placing the aliquot into a sampler's hand only for aliquot visualization purposes). Field experience has found that placing samples into a decontaminated 5-gallon bucket and then pouring the whole

sample into a bag is a better process. The likelihood of spilling increases with the use of a bag alone because, as the bag fills up, it is harder to eject additional soil increments into the bag. The bucket is more stable and may prevent loss of fines.

- Fill the holes left by sampling using surrounding soil or, if necessary, use sand to bring the subsurface sampling areas back to original grade. Do not include large rocks or pebbles in samples unless they are part of the overall soil matrix. It is not necessary to decontaminate the sampling tool between the increments within a single DU or SU.

- Obtain a pre-tared wide-mouth glass container and methanol from the analytical laboratory for sample aliquot preservation if collecting an ISM sample for volatile organic compound analysis. Collect increments using EnCore™ or TerraCore™ sampling tools. Note that a much smaller increment volume will be collected, resulting in a smaller total sample volume. Place smaller increments into the pre-tared glass containers containing methanol and follow the health and safety precautions associated with methanol handling. To prevent the loss of methanol through volatilization, keep the sample container closed as much as feasible and open only to place sample aliquots within the container.

- Collect the replicate samples from the same DU/SU following a different path, as shown in Figure SOP No. 057-1, although ITRC (2012) and other research have shown that replicate samples may be taken at the same time as the primary sample, by just using another corner of the grid, or off-stepping a meter or two. Establish the specific relative location of the replicate increments within each DU cell in a random manner to eliminate potential bias. To select the relative increment location for a replicate increment in a cell, divide the cell in turn into subgrids and select a subcell randomly generating a number on a calculator. Another selection method may be performed by rolling a dice for a 6 × 6 subgrid in the DU cell; the first die indicates the row and the second die the column of this subgrid.

- Label the large re-sealable bag containing the total sample volume with indelible ink. Follow the guidance listed under Section 5, Precautions below if methanol preserved sample containers will be shipped. Bubble-wrap and tape the sample(s) for shipping and place into iced coolers for transport under chain-of-custody protocol to the analytical laboratory. Follow the field procedure requirements set forth in EA SOP No. 002 – Chain-of-Custody Form and EA SOP No. 004 – Sample Packing and Shipping. Retain copies of the chain-of-custody forms and shipping documents in the project file. Document field activities according to logbook procedures specified in EA SOP No. 016 – Field Logbooks and Surface Water, Groundwater, and Soil/Sediment Field Checklists.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Safety precautions documented in the project-specific site Health and Safety Plan will be followed. If sampling procedures are to occur in areas where unexploded ordnance is known or potentially exist, the area will not be entered until unexploded ordnance support is provided. If an unsafe condition is identified, stop work immediately until the unsafe condition is mitigated. If sampling for volatile organic compound analysis, follow precautions associated with handling methanol. Also, because much larger quantities of methanol are employed for ISM sampling, follow the International Air Transport Association and Department of Transportation requirements associated with transportation of these samples. If shipped by air, excepted quantities are limited quantity (30 milliliters per inner package/receptable [i.e., sample container] and a total volume of no more than 0.5 liter per outer packaging [i.e., cooler]). Note that the outside of the sample shipment (cooler) should be labeled with excepted quantities marking.

6. REFERENCES

Interstate Technology and Regulatory Council (ITRC). 2012. *Incremental Sampling Methodology*. February.

U.S. Army Corps of Engineers. 2009. Interim Guidance 09-02 – Implementation of Incremental Sampling (IS) of Soil for the Military Munitions Response Program. Environmental and Munitions Center of Expertise Interim Guidance. 20 July.

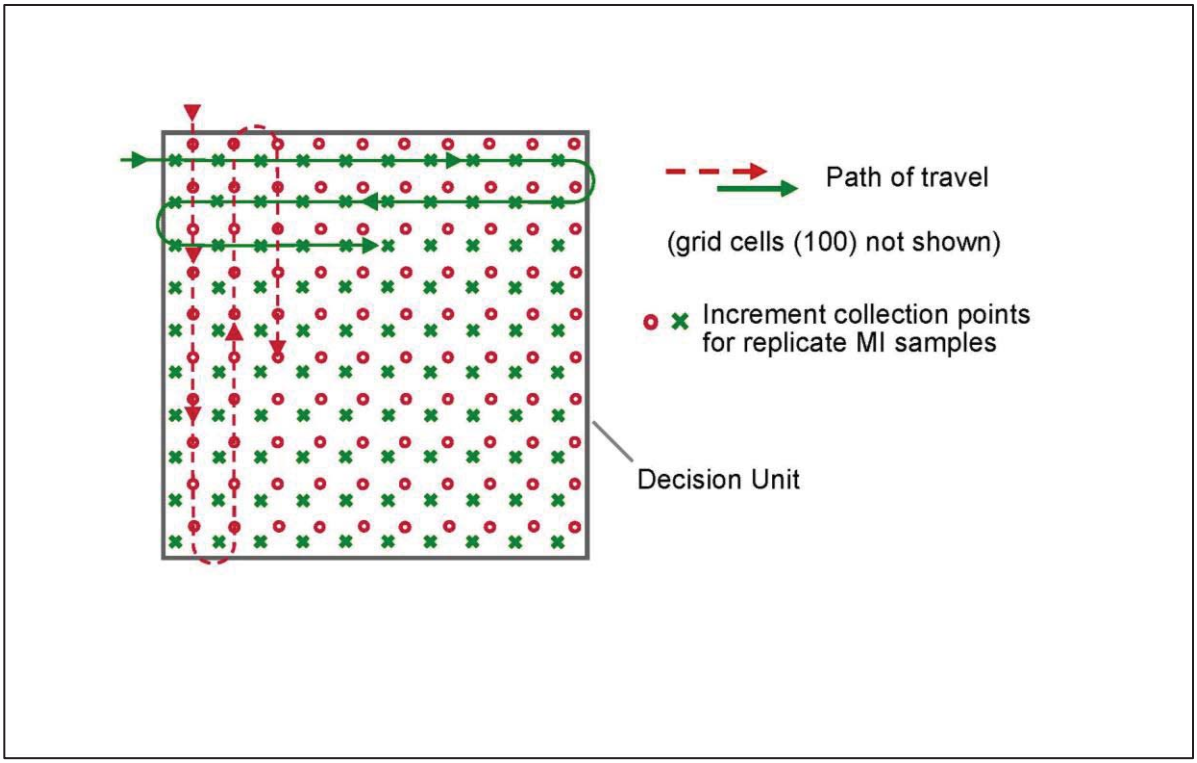

Figure SOP057-1. Example of Incremental Sampling in a Decision Unit.

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UFP-QAPP Addendum Appendix E

Analytical Standard Operating Procedures

Soil Processing Method ASTM D6323-12, ITRC Guidance Document, Soil Fractionation Method for Michigan	NC-OP-044 Rev 4
Subsampling, ASTM D6323-12	NC-OP-046 Rev. 3
Soil Processing – Ball Mill Procedure	WI-NC-0238_030521
Acid Digestion of Aqueous Samples for Metals Analysis by ICP	DV-IP-0010 Rev 13
Acid digestion of Aqueous Samples for Analysis by ICPMS (SW-846 3005A, 3020A, and EPA 200.8)	DV-IP-0014 Rev 12
Acid Digestion of Solids (EPA 3050B)	DV-IP-0015, Rev 14
PAHs by GC/MS SIM [SW 846 Method 8270C and 8270D]	DV-MS-0002 Rev 15
Mercury in Solids by Cold Vapor Atomic Absorption (CVAA) (SW-846 7471AB)	DV-MT-0016 Rev 14
Mercury in Solids by CVAA (SW-7470A)	DV-MT-0017 Rev 14
ICP MS for Trace Element Analysis by SW-846 Method 6020	DV-MT-0018 Rev 11
ICP Analysis for Trace Elements by SW-846 Method 6010C-D	DV-MT-0021 Rev 9
Incremental Sampling Methodology for Soils and Sediments (ASTM D 6323)	DV-OP-0013 Rev 13
Microwave Extraction of Solid Samples by Method [SW-846 3546]	DV-OP-0015
Colorimetric Sulfide in Waters (#0230), Sulfide as H2S (#10293 Calculation), Dissolved Sulfide in Water (#10499) by 4500-S2 D-2011, 4500-S2 F-2011, or EPA 376.2, Rev 17, effective 03/15/2018	WI11483
Acid Volatile Sulfide in Solids	TWCWI10422
Dilute/Run and AVS/SEM Sample Handling for Metals	TMETWI7920
Metals by ICP for Methods SW846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)	TMETWI11931
Grain Size Analysis, 06/20, Revision 3	CA-551-03
Titrimetric Determination of Organic Carbon Using the Walkley-Black Method, 05/13, Revision 0	CA-777-00
Sample Receipt and Internal Control, 05/20, Revision 14	SD-902-14
Sample Disposal, 09/17, Revision 6	SD-903-06
Cation-Exchange Capacity (CEC) of Soils	CC-ATM-M-20
CC-ATM-M005: Trace Metals Analysis by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)	CC-ATM-M005

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Title: Soil Processing

[Method: ASTM D6323-12, ITRC Guidance Document, Soil Fractionation Method for Michigan]

Approvals (Signature/Date):

Technology Specialist Diane Jones	Date 12/17/20	Health & Safety Coordinator Steve Jackson	Date 12/17/20
Quality Assurance Manager Mark J. Loeb	Date 01/11/21	Technology Director Raymond M. Risden	Date 12/18/20

This SOP was previously identified as NC-OP-044 Rev. 3, dated 12/05/18

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1. SCOPE AND APPLICATION

1.1. These disaggregation, mixing and particle size reduction techniques are applicable to a wide range soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, target analytes and quality objectives.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

Note: If foreign or quarantined solids are received, refer to SOP NC-SC-019 Canton Foreign Soils, current revision, and contact your Environmental Health and Safety Coordinator for proper handling instructions

2. SUMMARY OF METHOD

2.1. Air-Dried Soil Processing: The sample is air dried at room temperature and disaggregated to break clumps into a fine powder to facilitate obtaining a representative sub-sample and improving analyte extraction efficiency.

2.2. Soil Fractionation for Lead: Applicable to lead impacted soil and designed to distinguish the lead in the fine soil fraction from the coarse soil fraction.

2.3. Incremental Sample Wet Mixing Process: Applicable to mixing incremental samples into a single composite. These samples are NOT air dried, but rather water is added to facilitate mixing in a heavy-duty mixer.

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton QA Manual, current version.

3.2. Mix: To thoroughly blend the sample and reduce the analyte concentration differences between different parts of the overall sample. It is most effective when the particle size and density differences within the sample are small.

3.3. Incremental Sampling Methodology: A structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of the mean contaminant concentrations in an area/volume of soil targeted for sampling. ISM provides representative samples of specific soil areas/volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30-100 increments) that are combined, processed, and subsampled according to specific protocols.

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3.4. Grinding: A generic term for soil disaggregation or milling (ITRC Guidance Appendix E). The term grinding does not in itself represent any specific process.

3.5. Disaggregation: The act of breaking the soil clumps into individual small particles but keeping the small pebbles and hard crystalline particles intact (ITRC Guidance Appendix E).

3.6. Milling: Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. <250 µm or <75 µm) (ITRC Guidance Appendix E).

3.7. Sample: For laboratory technicians, the sample is all the material delivered to the laboratory in a container collected by the field crew.

3.8. Subsample: The small representative amount removed from a field sample that selected for final analysis. Also referred to in some SOPs as “the aliquot”.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants (i.e. samples being analyzed for VOCs should generally not come into contact with equipment that has been solvent-rinsed with acetone or methylene chloride).

4.2. Metallic components of particle size reduction equipment can contribute some metal content to the solid samples. Hence carbon steel components are usually preferable to stainless steel when necessary to minimize contamination from chromium, nickel and molybdenum. Some contamination from iron is common.

4.3. Particle size reduction equipment blanks should be generated before use. The type of blank generated may vary for each piece of equipment, and may be solid or aqueous in nature.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the NDSC EH&S Manual, the Facility Addendum to the NDSC EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn while doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Overexposure may include headache, drowsiness and dizziness. Methanol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur: symptoms may be parallel to inhalation exposure. Irritant to the eyes.
Hydrochloric acid	Corrosive Poison	5ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.6. When operating the electric chopper or grinder, be sure to keep all aqueous liquids clear to prevent the risk of electrical shock from any spills.

5.7. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection. The Environmental Health and Safety Coordinator must approve any and all use of dust masks and/or respirators.

5.8. If there is any malfunction in the equipment de-energize and tag out.

5.9. All noise levels are below OSHA limits.

5.10. Training: Only trained personnel are permitted to use the equipment mentioned in this document. A list of trained personnel will be maintained with EH&S and QA.

6. EQUIPMENT AND SUPPLIES

6.1. Drying tray, plastic or aluminum

6.1.1. Half cake sheet pan, Pactiv #614255, or equivalent

6.1.2. Quarter sheet cake pan, Pactiv #604245, or equivalent

6.2. Butcher Paper

6.3. Plastic wrap

6.4. Mortar and pestle

6.5. Automated mortar grinder

6.6. Food chopper, Black and Decker Handi chopper, or DeLonghi mini food processor, or equivalent

6.7. Coffee grinder, KitchenAid BCG100 or equivalent

6.8. Automated soil disaggregator

6.9. Wooden spatula: 6 in.

6.10. Stainless steel sieves: 1 mm, #10, #20, #60, #100, ¼ inch, other sizes by request with advanced notice

6.11. Electrolux Assistant 8 qt mixer, with dough hook or equivalent

6.12. Large cookie scoop with hand actuated ejector blade

6.13. Small cookie scoop with hand actuated ejector blade

6.14. Fluoropolymer scoop, not commercially available

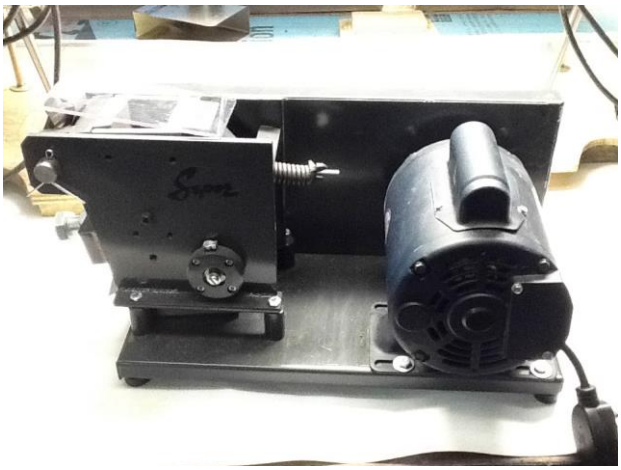
6.14.1. Construct by fastening the bottom half of a fluoropolymer bottle to a wooden handle with stainless steel screws.

6.15. Stainless steel pot with cover, Bain-Marie 6 qt. or equivalent

6.16. Wooden spoon, 12” long

Picture of labware for multi-increment sample wet mixing process (Section 11.3.2.5): large scoop, small scoop, wooden spoon, fluoropolymer scoop, stainless steel pot/cover

6.17. Jaw crusher, Sepor Model 150 or equivalent



6.18. Freezer for sample storage.

6.19. 8 oz. Glass jars with lids

6.20. Top loading balance capable of weighing 100 g ±0.2 g.

6.21. Aluminum foil

7. REAGENTS AND STANDARDS

7.1. Deionized water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

7.2. Methanol: VOC grade

7.3. Acetone: Pesticide grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Not applicable to this procedure. Sample collection, preservation, and storage are dependent on the requested test method and analytes.

9. QUALITY CONTROL

9.1. Equipment processing blanks might be applicable for some particle size reduction or selection techniques. There is no single blank matrix that is suitable for all analytes, equipment or processes. The blank matrix should be chosen by the client/data user based on the advantages and limitations described below in Section 9. If no blank

matrix is selected by the client, reagent water should be used as the default when possible as noted in Section 9.2.

9.2. Reagent water (or organic solvents) may be used to rinse the equipment surfaces. This liquid is then analyzed for the target analytes. This process is good at monitoring residue on equipment surfaces from previously processed samples. It does not evaluate the potential contribution of the equipment surface material to a solid sample. Sand may be run through processing equipment and then analyzed to monitor for both sample carryover residue and contamination from equipment surface erosion. This process is most applicable for organic analytes. Sand always contains metals. These metals concentrations might be too high for suitable blank demonstrations. Also, the sand material is frequently more abrasive than soil and can over-estimate sample contamination due to erosion of the equipment surfaces.

9.3. Teflon boiling chips can be suitable to monitor the cleanliness of processing equipment surfaces. This option is between reagent water and sand regarding abrasion of the equipment surfaces. This material is generally non-detect at parts per billion concentrations for most organic and inorganic analytes. The Teflon material does not mimic the behavior of soil in the subsequent sample extraction or digestion procedures.

9.4. As an alternative to or in addition to equipment blanks, half of a field sample may be reprocessed with the goal of evaluating differing analyte concentrations between the single and double processed sample aliquots. The occurrence of significantly elevated analyte concentrations in the double processed sample can be a good indication of carryover or contamination due to equipment surface erosion. This is most applicable for monitoring metals contamination in soil grinders.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable to this procedure.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. The following sections describe a variety of procedures. The client/data user must select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives. The selection of the procedure(s)

can be made by the client in consultation with the appropriate laboratory representatives during project planning. There is no single default procedure that can be used in the absence of client selection.

11.3.2. Solid Sample Mixing and Particle Size Reduction Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wood blade.

11.3.2.3. Mortar and pestle

11.3.2.3.1. A mortar and pestle can be used for samples in the 1 to 8 mm particle size. The final reduced size is between 5 um and 8 mm. This process can be used for wet or dry, organic or inorganic substances.

11.3.2.3.2. Fill the mortar about 1/3 with sample. Grind and mix the sample with the pestle. Transfer the processed sample to a separate container, and repeat the grind-and-mix step with additional sample aliquots as needed.

11.3.2.3.3. An automated mortar grinder may be appropriate for larger samples. Set the automated mortar grinder to the desired size and allow to run until the sample has been completely ground. If the grinder is not equipped with an automated scraper/agitator, the sample may be mixed by hand. Be sure that the grinder has stopped moving completely before opening the lid.

11.3.2.4. Dry mixing (incremental sampling methodology support)

11.3.2.4.1. Air dry, disaggregate, sieve, mix

11.3.2.4.1.1. Line a standard aluminum tray with a disposable aluminum tray ($^1/_2$ or $^1/_4$ sheet depending on sample size). If aluminum is a metal of interest line the tray with butcher paper. Do not use butcher paper when TOCs are of interest.

11.3.2.4.1.2. Remove large rocks and vegetation unless client indicates otherwise. Do not decant the free water. Mix the sample and spread the entire sample in a thin layer in the tray. If necessary to perform a pre-dried total solids analysis, remove approximately 10 g. If the samples require ISM processing, an appropriate subsampling procedure should be followed, as described in the subsampling SOP.

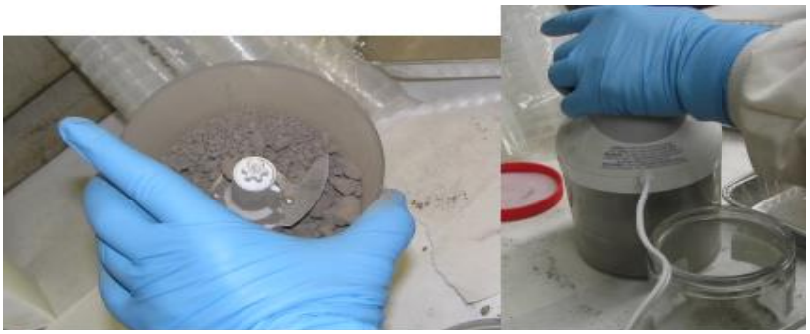
Note: it is not recommended to remove any portion of an ISM sample prior to drying and disaggregation. Any such removal from an ISM sample is considered a deviation and should be approved by the client.

11.3.2.4.1.3. Place the tray in the ventilated drying rack for up to five days at room temperature.

11.3.2.4.1.4. Periodically, stir the sample to expose moist sample to the air.

11.3.2.4.1.5. The dried sample must be crushable, and not prone to sticking together. Typically this means that sediment samples will be less than 30% moisture content and soil samples less than 15% moisture content. However, it is not necessary to do a percent moisture analysis.

11.3.2.4.1.6. Disaggregate the dried sample to break up the dried sample clumps with a bladed chopper or equivalent, but do not grind the small pebbles into powder.



11.3.2.4.1.7. If the volume capacity of the disaggregation equipment is large enough, transfer the whole sample to the disaggregator. If sample volume is too large, disaggregate the sample in sub-aliquots.

11.3.2.4.1.8. Pass the sample through a sieve to remove small pebbles and organic materials. Project specific guidelines will determine the sieve opening size. Typically, a #10 sieve is used for soils.

11.3.2.4.1.9. Combine all disaggregated sub-aliquots, and mix thoroughly by stirring, shaking or tumbling.

11.3.2.4.2. Air dry, disaggregate, mix

11.3.2.4.2.1. When the project objectives dictate that all small and medium sized materials be included in the final sample, use the procedure described in Section 11.3.2.4.1, but skip the sieving step in Section 11.3.2.4.1.8.

11.3.2.4.2.2. Collecting a representative subaliquot is more difficult when the sample has a variety of particle sizes. Refer to the subsampling SOP for recommended procedures. The one dimensional slabcake process is particularly applicable to dry, flowable samples with a wide range of particle sizes.

11.3.2.4.3. Other

11.3.2.4.3.1. Various permutations of soil disaggregation and sieving are possible depending on the needs of the project and can be accommodated but must be clearly defined in consultation with the client. These variations can include different sieve sizes and changing the order so that sieving is done prior to disaggregation. This would most likely be done to exclude a particular size of organic or rocky material.

11.3.2.5. Wet mixing (incremental sampling methodology support)

11.3.2.5.1. Weigh 100.0 +/- 0.2 g of each ¼ acre sub-sample. Stir the sample prior to and during the transfer from the original container to the weighing container. Record the weight to two decimal places.

11.3.2.5.1.1. If there are fewer than 40 sub-samples, increase the weight of sample proportionally. Calculate the new target weight as follows: target weight = 4000g/# sub-samples.

11.3.2.5.1.2. Exclude rocks, organic matter, and other debris from the weighed sub-sample by sieving through a ¼ inch sieve prior to weighing when such material is present.

11.3.2.5.1.3. The sieve may also be used to break up clay chunks. Sieving may be done either before or after weighing for this purpose. Use only when needed. Hard dry soil agglomerates should be broken up by hand crushing or chopping with a food chopper.

11.3.2.5.1.4. If a sub-sample is over ½ gravel, expand the maximum particle size from ¼ inch to 1.8 inches.

11.3.2.5.2. Transfer the weighed (and sieved as needed) sub-sample aliquot to a stainless steel pot used to collect all weighed aliquots for that composite sample. The total mass of the composite sample will be at least 4 kg.

11.3.2.5.3. Repeat Section 11.3.2.5.1 until all sub-samples have been weighed and transferred to the compositing pot.

11.3.2.5.4. Assemble the heavy-duty mixer with mixing hook. The mixing hook must be inserted high in the mounting bracket to avoid dragging on the bottom of the bowl and allow small stones to pass under the hook. Transfer the entire composite sample from the covered stainless steel pot to the stainless mixing bowl.

11.3.2.5.5. Turn on mixer. Add reagent water to ensure complete mixing of the sample. The mixed sample will have uniformly distributed water, look visually homogeneous, and have the consistency of a thick paste. Do not add so much water as to form runny slurry. Use a wooden spoon to assist the mixing process by scraping mud from the sides and directing it to the center. The picture below shows the proper consistency. Mix for three minutes after the proper consistency has been achieved.

completely mixed composite sample.mpg

11.3.2.5.6. Record the volume of reagent water added and the total mixing time.

11.3.2.5.7. Scrape the mud from the wooden spoon and mixing hook.

11.3.2.5.8. Split the composite sample between 5-15 250 mL jars, depending on project requirements.

11.3.2.5.9. Use the large scoop to dispense the equal aliquots of the composite sample--one aliquot into each of 8 oz. jars. Repeat the process if there is sufficient sample for a complete set of large scoop aliquots (per Section 11.3.2.5.8).

large scoop use.mpg

11.3.2.5.10. Once the volume of wet composite sample in the bowl is less than the determined set of large scoops, switch to aliquoting with the small scoop. Dispense an equal amount of small scoops--one into each 8 oz. jar.

11.3.2.5.11. Use the fluoropolymer scoop to scrape inside the bowl, and dispense this part of the sample with the small scoop. If

there is insufficient sample to use full scoops, the use of replicate partial scoops is acceptable.

11.3.2.5.12. When aliquoting is complete, the composite sample has been evenly distributed among the 8 oz. jars. Note: Multiple jars are used for various analyses, quality control, and archive purposes.

11.3.2.5.13. Mix the contents of each 250 mL jar and remove about 10 g from each of the first three jars for three total solids (percent moisture) analyses. The relative percent difference (RPD) between the first two must not exceed 8%.

11.3.2.5.13.1. If the RPD exceeds 8%, repeat the total solids analysis on two fresh aliquots from the first two jars.

11.3.2.5.13.2. If the RPD still exceeds 8%, repeat the mixing process in Sections 11.3.2.5.1 to 11.3.2.5.12

11.3.2.5.14. Wipe the top of the jar to remove excess sample and install the cap. Transport all sample containers to Sample Receiving.

11.3.2.5.15. Discard the wooden spoon and spatulas. Wash the mixer bowl, hook and scoops using soap and water. Rinse with tap and reagent water.

11.3.2.6. Soil fractionation for lead analysis

11.3.2.6.1. Add sample ID label to outside of a disposable aluminum ($^1/_2$ or $^1/_4$ sheet depending on sample size). Line with butcher paper if aluminum is also an analyte of interest.

11.3.2.6.2. Transfer the soil sample to a tray. Remove large rocks and vegetation. Spreading the sample in a thin layer speeds drying and reduces the formation of hard clay chunks.

11.3.2.6.3. Place the tray in the ventilated drying rack for up to five days.

11.3.2.6.4. Periodically, stir the sample if needed to expose moist sample to the air. The butcher paper may also be changed, if it has become damp.

11.3.2.6.5. Samples with high clay content tend to form large, hard clay aggregates. To reduce the formation of these hard “bricks”, use the bottom of a clean disposable beaker to gently crush the semi-dried sample before drying is complete.

11.3.2.6.6. Allow the sample to dry until crumbly.

11.3.2.6.7. Assemble 8-inch sieve stack. The order from the bottom of the stack is collection pan, #60 sieve, and #10 sieve. Transfer sample ID label from drying pan to sieve stack.

Note: Project-specific sieve size requirements may vary from the reference method. These will be noted in LIMS, both in the project and in the method or login notes. #100 (150um) sieves are commonly used in place of #60 sieves. The smallest sieve size should be the closest to the collection pan, with the next size up above it.

11.3.2.6.8. If significant amounts of clay are present, aggregates may be broken by pressing gently with a hand-operated mortar and pestle. Note that only this gentle crushing is permitted by the reference method. Samples that are being analyzed as soil fractions for lead should not be subjected to aggressive disaggregation such as the use of a soil disaggregator or coffee grinder.

11.3.2.6.9. Transfer dried soil to the #10 sieve at the top of the sieve stack. If fine particulates are present, use sufficient ventilation to prevent the analyst from inhaling the dust. Install sieve cover.

11.3.2.6.1. Place the sieve stack on the shaker table and secure. Run the shaker for one hour. Stop the shaker and remove the lid for each sample to check on the progress of sieving. Shake the samples by hand (in a different direction than the shaker table has traveled). If any sample passes through the sieve at this point, place the sieve stack(s) back on the shaker and shake for up to one additional hour.

11.3.2.6.2. Do not extend shaking beyond two hours without conferring with the client.

11.3.2.6.3. Record the weights of the three fractions--large (did not pass through the #10 sieve), coarse (passed through #10 sieve, but not the #60 sieve), and fine (passed through both #10 and #60 sieves). If different sieve sizes have been used, record the sizes on the bench sheet.

11.3.2.6.4. To weigh each fraction, place the corresponding sieve or pan on the balance, press “Tare”, transfer the fraction, and place sieve or pan back on the balance. The weight of the fraction will read as a negative value.

11.3.2.6.5. Discard the large fraction. Mix the fine fraction. Remove approximately 10g for total solids analysis. If required, perform splitting and/or subsampling in accordance with ISM procedures. See the subsampling SOP for further information.

11.3.2.6.6. If the coarse fraction is to be analyzed separately, mix the coarse fraction and remove approximately 10g for total solids analysis. The coarse fraction should be ground in a mortar and pestle (Section 11.3.2.3) prior to removing the aliquot for metals digestion.

11.3.2.6.7. The combination of coarse and fine fractions is defined as the “total”. If the “total” is to be measured, weigh 1/10 of the coarse fraction and 1/10 of the fine fraction. Combine, mix, and remove approximately 10g for total solids analysis.

11.3.2.6.8. Bottle coarse, fine, and “total”(if needed) fractions for metals analysis.

11.3.2.6.9. For Michigan samples, the “total” result is calculated from a weighted average of the results from the fine and coarse fractions.

Total Lead = [(A x Wf) + (B x Wc)] / (Wf + Wc)

Where:

A = Concentration of Lead (mg/Kg dry) in fine fraction

B = Concentration of Lead (mg/Kg dry) in coarse fraction

Wf = Total weight of fine fraction

Wc = Total weight of coarse fraction

11.3.2.6.10. Wash the sieves with soap, tap water, and deionized water. Dislodge objects from the screen with a green scratch pad, wooden tongue blade or small screwdriver, as necessary. Dry sieves in a low-heat oven or air-dry at ambient temperature over night, depending how soon they will be needed.

11.4. Sample Analysis

11.4.1. Not applicable to this procedure.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001 for "Waste management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001 for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.

15.2.2. Dry ice is to be melted in the sink.

15.2.3. Acetone is to be disposed of in an appropriately labeled flammable waste barrel.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Eurofins TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. U.S. EPA, 2000. TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites. EPA-540-F-00-010. OSWER 9285.7-38. April. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/sssiev.pdf

16.1.2. U.S. EPA, 2003. TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges. OSWER 9285.7-37. March. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/firing.pdf

16.1.3. U.S. EPA, 2003. Superfund Lead-Contaminated Residential Sites handbook. OSWER 9285.7-50. August. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/handbook.pdf

16.1.4. Michigan DEQ SOP #213 Revision #1, Nov. 9, 2004, Soil Fractions Preparation for Lead Analysis (Creating Total, Fine and Coarse Soil Samples).

16.1.5. ASTM D 6323-12, Laboratory Subsampling of Media Related to Waste Management Activities, 2012

16.1.6. ITRC Incremental Sampling Methodology, Available online at: http://www.itrcweb.org/ism-1/

16.1.7. Eurofins TestAmerica Canton QA Manual, current version

16.1.8. NDSC EH&S Manual Document No. CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9. Quality Management Plan (QMP), current version

16.1.10 Revision History

Historical File:				
Revision 0: 11/27/13				
Revision 1: 01/23/15				
Revision 2: 05/31/17				
Revision 3: 12/5/18				

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Not applicable to this procedure

17.2. Method deviations – None

Title: SUBSAMPLING

[Method: ASTM D6323-12]

Approvals (Signature/Date):

Technology Specialist	05/15/19 Date	Health & Safety Coordinator	05/16/19 Date
Quality Assurance Officer	06/18/19 Date	Technical Director	05/29/19 Date

This SOP was previously identified as SOP No. NC-OP-046, Rev 2, dated 5/31/17

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1. SCOPE AND APPLICATION

1.1. These mixing, subsampling, and splitting techniques are applicable to a wide range of soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, target analytes and quality objectives.

1.2. The goal of all mixing, subsampling, and splitting techniques is to obtain representative splits or subsamples for preparation and analysis. Particle size reduction and processing can be beneficial in obtaining representative subsamples and specific processes for particle size reduction and processing can be found in the soil processing SOP (NC-OP-044) and non-soil processing SOP (NC-OP-045).

1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

NOTE: If foreign or quarantined solids are received, refer to SOP NC-SM-019 Canton Foreign Soils, current revision, and contact your Environmental Health and Safety Coordinator for proper handling instructions.

2. SUMMARY OF METHOD

2.1. All samples should be mixed and subsampled appropriately for the test and analytes of interest.

2.2. Solid sample mixing procedures include in-jar mixing, horizontal surface mixing, and mortar and pestle.

2.3. Solid sample subsampling procedures include alternate scoop, one-dimensional slabcake, two-dimensional slabcake, and cone and quarter methods.

2.4. Liquid sample subsampling procedures include centrifuge, pipettes, coliwasa devices, and multiphase procedures.

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version.

3.2. Mix: To thoroughly blend the sample and reduce the analyte concentration differences between different parts of the overall sample. It is most effective when the particle size and density differences within the sample are small.

3.3. Incremental Sampling Methodology: A structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of the mean contaminant concentrations in an area/volume of soil targeted for sampling. ISM

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provides representative samples of specific soil areas/volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30-100 increments) that are combined, processed, and subsampled according to specific protocols.

3.4. Grinding: A generic term for soil disaggregation or milling.

3.5. Disaggregation: The act of breaking the soil clumps into individual small particles but keeping the small pebbles and hard crystalline particles intact.

3.6. Milling: Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. <250 µm or <75 µm).

3.7. Sample: For laboratory technicians, the sample is all the material delivered to the laboratory in a container collected by the field crew.

3.8. Subsample: The small representative amount removed from a field sample selected for final analysis. This is also referred to in some SOPs as the aliquot.

3.9. Representative subsample: A subsample taken in such a way that each particle had an equal chance of being selected. The most representative subsample is one that most closely resembles the true value of the material sampled or contains the least amount of error.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Metallic components of particle size reduction equipment can contribute some metal content to the solid samples. Hence carbon steel components are usually preferable to stainless steel when necessary to minimize contamination from chromium, nickel and molybdenum. Some contamination from iron is common.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.4. There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the Safety Data Sheet (SDS) for each material prior to using it for the first time, or when there are major changes to the SDS.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.6. When operating the electric chopper or grinder, be sure to keep all aqueous liquids clear to prevent the risk of electrical shock from any spills.

5.7. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection.

5.8. If there is any malfunction in the equipment. De-energize and tag out.

5.9. All noise levels are below OSHA limits.

5.10. Only trained personnel are permitted to use crushing equipment. A list of trained personnel will be maintained with EH&S and QA.

6. EQUIPMENT AND SUPPLIES

6.1. Butcher Paper

6.2. Plastic wrap

6.3. Aluminum foil

6.4. 8 oz. Glass jars with lids

6.5. Top-loading balance.

6.6. Appropriate containers for each subsample

7. REAGENTS AND STANDARDS

7.1. Deionized water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Not applicable to this procedure. Sample collection, preservation, and storage are dependent on the requested test method and analytes.

9. QUALITY CONTROL

9.1. Equipment processing blanks might be applicable for some particle size reduction or selection techniques. There is no single blank matrix that is suitable for all analytes, equipment or processes. The blank matrix should be chosen by the client/data user based on the advantages and limitations described below in Section 9. If no blank matrix is selected by the client, reagent water should be used as the default when possible, as noted in Section 9.2.

9.2. Reagent water (or organic solvents) may be used to rinse the equipment surfaces. This liquid is then analyzed for the target analytes. This process is good at monitoring residue on equipment surfaces from previously processed samples. It does not evaluate the potential contribution of the equipment surface material to a solid sample.

9.3. Sand may be run through processing equipment and then analyzed to monitor for both sample carryover residue and contamination from equipment surface erosion. This process is most applicable for organic analytes. Sand always contains metals. These metals concentrations might be too high for suitable blank demonstrations. Also, the sand material is frequently more abrasive than soil and can over estimate sample contamination due to erosion of the equipment surfaces.

9.4. Teflon boiling chips can be suitable to monitor the cleanliness of processing equipment surfaces. This option is between reagent water and sand regarding abrasion of the equipment surfaces. This material is generally non-detect at parts per billion concentrations for most organic and inorganic analytes. The Teflon material does not mimic the behavior of soil in the subsequent sample extraction or digestion procedures.

9.5. Reprocess half of a field sample and look for differing analyte concentrations between the single and double processed sample aliquots. The occurrence of significantly elevated analyte concentrations in the double processed sample can be a good indication of carryover or contamination due to equipment surface erosion. This is most applicable for monitoring metals contamination in soil grinders.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable to this procedure.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. The following sections describe a variety of procedures. Additional procedures for sample preparation are available in the Soil and Non-Soil Processing SOPs. The client/data user must select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives. The selection of the procedure(s) can be made by the client in consultation with the appropriate Eurofins TestAmerica representatives during project planning. There is no single default procedure that can be used in the absence of client selection.

11.3.2. Solid Sample Mixing Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wooden blade.

11.3.3. Solid Sample Subsampling Procedures

11.3.3.1. Multiple increments from a jar

11.3.3.1.1. Select 5-10 small sample increments from various locations within the sample container to make the entire subaliquot. The increments must come from all general areas of the container--top, sides, center, and bottom.

11.3.3.2. Two dimensional slabcake

11.3.3.2.1. Spread the sample to a consistent depth on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum tray or foil or plastic depending on the analytes of interest).

11.3.3.2.2. Select 30 or more small increments spread evenly over the sample area or as dictated by client data quality objectives.

The increments must be collected with a blunt end spatula or scoop to evenly collect from the top middle and bottom of the slabcake. A coring device of suitable material can also be used if the sample is sufficiently cohesive (e.g. moist soil).

11.3.3.2.3. If the sample has noxious odors or produces dust, the sample spreading and increment collecting should be performed in a hood or large flat bag of appropriate material. The analyst must be protected from potential inhalation hazards from the sample.

11.3.3.3. Alternate scoop

11.3.3.3.1. Wet or dry solid samples can be divided into two or more smaller aliquots. Determine the number of sub-aliquots, and prepare that many empty containers.

11.3.3.3.2. Scoops of the mixed laboratory sample are either placed in the analytical vessel or discarded.

11.3.3.3.3. Three scoops are discarded for every scoop saved. Randomly select an aliquot and place in the first container. Place the next three aliquots in the second container. Repeat as needed for additional aliquot containers.

11.3.3.3.4. Repeat the aliquoting cycle until the original sample is consumed.

11.3.3.3.5. The alternate scoop technique can also be used to subaliquot discrete samples that are to be homogenized into one composite sample.

11.3.3.3.5.1. Determine approximately the ratio of aliquot to discard scoops required to consume the sample and achieve the aliquot size required for homogenization. For example, if a 50 g aliquot is desired from a 250 g sample, a ratio of one scoop used to four scoops discarded will be used.

11.3.3.3.5.2. For each discrete sample, set up two empty containers of appropriate volume.

11.3.3.3.5.3. Stir each discrete sample in its container, or if necessary homogenize on a sheet of butcher paper and return to the jar.

11.3.3.3.5.4. Place one of the empty containers on a balance and tare it. Place one scoop of sample from the top

of the discrete sample jar into this container. This will be the portion used for a homogenized composite later.

11.3.3.3.5.5. Into the second jar, place the three or more (as determined in Section 11.3.3.3.5.1) scoops to be discarded.

11.3.3.3.5.6. Repeat Sections 11.3.3.3.5.4 and 11.3.3.3.5.5 until the sample has been consumed and the desired weight of the subaliquot is in the tared container. Record the weight of the sub-aliquot.

11.3.3.3.5.7. Repeat this process for all of the discrete samples that will be used for the composite, using approximately equal weights of each discrete sample.

11.3.3.3.5.8. After all discrete samples have been sub-aliquoted using the alternate scoop procedure, combine them on a sheet of butcher paper and homogenize. Pour the homogenized sample volume into the labeled sample jar for the composite sample.

11.3.3.4. One dimensional slabcake

11.3.3.4.1. This process is intended to produce large sub-samples from very large dry flowable solid samples, such as those collected using \incremental sampling methodology, dried and then chopped using the process described in Section 1.1.1See Reference 16.1.7.

11.3.3.4.2. Pour the dry soil sample into a long thin pile onto a clean horizontal surface. The pour height should not exceed 20 cm to minimize the formation of a dust cloud.

11.3.3.4.3. Ensure that the sample container makes at least 20 passes back and forth over the “line of sample”.

11.3.3.4.4. Using a rectangular flat-bottomed scoop, remove an increment from the “line of sample”. Ensure that a complete cross cut of the sample line includes the entire depth of that increment. Combine increments as needed to produce the needed sub-sample size.

11.3.3.5. Cone and quarter (for dry flowable samples)

11.3.3.5.1. Pour the sample into a cone on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum foil, or plastic depending on the analytes of interest).

11.3.3.5.2. Cut the cone in half in two directions to form four quadrants.

11.3.3.5.3. Return opposite quadrants to the original sample container.

11.3.3.5.4. Repeat the process in Sections 11.3.3.5.1 through 11.3.3.5.3 until the needed aliquot size has been obtained.

11.3.4. Liquid Sample Mixing Procedures

11.3.4.1. Closed container shaking

11.3.4.1.1. All liquid samples should be mixed by shaking in the original closed sample container unless a multiple layer sub-aliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be sub-aliquoted using an appropriate technique as described below.

11.3.4.1.2. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.5. Liquid Sample Subsampling Procedures

11.3.5.1. Pour

11.3.5.1.1. Samples that are well mixed can be sub-aliquoted by pouring an appropriate volume off the top of the sample.

11.3.5.2. Layer Subsampling

11.3.5.2.1. Some liquid samples with multiple layers separate too quickly to pour a representative sub-aliquot off the top. In some instances it is best to separate the layers and handle them as individual samples. In other instances representative aliquots of each layer must be collected and processed as a single sub-sample.

11.3.5.2.2. Unless directed otherwise, record the phase ratios (or volumes) of the layers.

11.3.5.2.3. Gravity or Centrifuge Settling

11.3.5.2.3.1. Allow the sample layers to separate based on density. Centrifugation can be used to accelerate the process if simple gravity settling is not fast enough.

11.3.5.2.4. Separatory Funnel

11.3.5.2.4.1. Gently pour the sample into a separatory funnel. Allow time for additional layer separation as needed.

11.3.5.2.4.2. Drain the layers out into separate containers one at a time.

11.3.5.2.4.3. Some oils stick to the separatory funnel sides so the draining process must be slow enough to avoid remixing the layers.

11.3.5.2.5. Pipette

11.3.5.2.5.1. Use a pipette of appropriate size to collect a sub-aliquot of a sample layer of interest and transfer to an empty container.

11.3.5.2.5.2. If the entire sample is to be separated, pipette almost all the top layer into a new container. Draw the small volume of the top layer into the pipette along with a small portion of the second layer. Allow the two layers to separate in the pipette (like a separatory funnel). Dispense the bottom layer back into original sample container with the bulk of the bottom layer. Next dispense the remaining top layer into the container that holds the top layer.

11.3.5.2.5.3. Repeat as needed for each layer of interest.

11.3.5.2.6. Coliwasa

11.3.5.2.6.1. A coliwasa is a “coring device” designed for liquid multi-layer samples. It is a long tube with a short valve at the bottom. When designed and used properly it collects representative aliquots of each layer with each sub-aliquoting immersion.

11.3.5.2.6.2. The volume collected is determined by the diameter of the coliwasa and the height of the sample. The larger the tube diameter or taller the sample height, the larger the volume of sample collected.

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11.3.5.2.6.3. Place the foot of the valve and its control rod in the multi-layer sample. Slowly slide the coliwasa tube over the control rod and close the ground glass seal of the valve at the bottom.

11.3.5.2.6.4. Lift the coliwasa by the control rod to keep the valve sealed.

11.3.5.2.6.5. Place the coliwasa over the new sample aliquot container. Grasp the top of the coliwasa tube and slowly lower the control rod a few millimeters to open the foot valve and drain the sample into the container.

11.3.5.2.6.6. Repeat Sections 11.3.5.2.6.3 through 11.3.5.2.6.5 until sufficient sample aliquot has been collected.

11.3.6. Multiphase Sample Mixing Procedures

11.3.6.1. All liquid samples should be mixed by shaking unless a multiple layer subaliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be subaliquoted using an appropriate technique as described below.

11.3.6.2. Closed container shaking

11.3.6.2.1. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.6.3. Open container stirring

11.3.6.3.1. The sample is stirred or swirled to mix.

11.3.6.4. Wet Sediments

11.3.6.4.1. Aqueous samples for non-volatile compound analysis may contain settleable materials. If the settleable materials are to be included as part of the laboratory sample, and they will remain suspended, or can easily be re-suspended and will remain so during the subsampling operation, the sample should be handled as a liquid sample. These samples should be gently swirled for 15 seconds or slowly inverted six times to reduce heterogeneity.

11.3.6.4.2. If the liquid portion only is to be used, the settleable material must be allowed to sink to the bottom before withdrawing the subsample.

11.3.6.4.3. If the settleable material will not remain suspended and is to be included in the analysis, the sample should be treated as a multilayered sample.

11.3.7. Multi-phase Sample Subsampling Procedures

11.3.7.1. Multi-layered samples may include liquid/liquid layers, liquid/solid samples, or solid/solid samples.

11.3.7.2. If the liquid portion of a sludge sample can be re-mixed with the solid portion and will re-separate over time, the sample should be handled as a solid sample.

11.3.7.3. If the solid portion will remain suspended or can easily be re-suspended, the sample should be treated as a liquid sample. Mixing may occur by inverting the container or with slight shaking

11.3.7.4. Layer Separating

11.3.7.4.1. Gravity/Centrifuge – The solid/liquid phase separation may be achieved by gently centrifuging the unopened container or by allowing it to sit undisturbed until the solid portion is settled.

11.3.7.4.2. Pipette – For liquid/liquid layers, separate the layers using a pipette. Each layer may be either transferred into another container or directly into the analytical vessel. Each separated portion is then handled as a homogeneous liquid sample.

11.3.7.4.3. Filter/Decant – For liquid/solid layers, the liquid subsample may be obtained by filtering or decanting the liquid portion from the solid portion. The liquid portion is then handled as a homogeneous liquid sample. The solid portion is handled as a solid laboratory sample.

11.4. Sample Analysis

11.4.1. Not applicable to this procedure

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Eurofins TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. U.S. EPA, 2000. TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites. EPA-540-F-00-010. OSWER 9285.7-38. April. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/sssiev.pdf

16.1.2. U.S. EPA, 2003. TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges. OSWER 9285.7-37. March. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/firing.pdf

16.1.3. U.S. EPA, 2003. Superfund Lead-Contaminated Residential Sites handbook. OSWER 9285.7-50. August. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/handbook.pdf

16.1.4. Michigan DEQ SOP #213 Revision #1, Nov. 9, 2004, Soil Fractions Preparation for Lead Analysis (Creating Total, Fine and Coarse Soil Samples). Available on-line at: http://www.deq.state.mi.us/documents/deq-rrd-OpMemo_2_SoilFractionsPrepForLead.pdf

16.1.5. ASTM D 6323-12, Laboratory Subsampling of Media Related to Waste Management Activities, 2012

16.1.6. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.7. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.8. Corporate Quality Management Plan (CQMP), current version

16.1.10 Revision History

Historical File:		Revision 0: 11/27/14		
		Revision 1: 01/23/15		
		Revision 2: 05/31/17		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Not applicable to this procedure

17.2. Method deviations – None

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Soil Processing – Ball Mill Procedure

Eurofins TestAmerica Canton Soil Processing SOP NC-OP-044 offers optional procedures to meet project specific requirements. This work instruction details the steps followed for Ball Milling.

1. Start with dried sample as specified in the Drying section of the Soil Processing SOP.

2. Print labels for the paint can and affix to sides and tops of each can. The sample ID may also be written in permanent marker if necessary.

3. Load dried sample into can.

   3.1. Do not fill the paint can over halfway full. If the can is filled too much, inefficient milling will occur.

   3.2. Multiple paint cans may be necessary. Label each can appropriately (ie. Sample ID and 1 of 2, 2 of 2).

4. Place 3-5 large pieces of grinding media into the paint can, this will depend on the amount of sample.

5. Seal the lid onto the paint can with a mallet.

6. Secure lid with duct tape.

7. Place a “dummy” paint can on the edges closest to the control panel. Make sure that this can is well-sealed and has sand and media for weight.

8. Start the ball mill.

9. Record the dates, times, and analyst’s initials in the batch.

10. Each sample should roll on the ball mill overnight, or for a minimum of 8 hours.

11. At the end of the processing cycle, turn off the ball mill and remove the paint cans from the ball mill.

12. With proper ventilation, open the paint cans and visually inspect the sample to ensure that the sample has been adequately milled. Once the sample is adequately milled, remove the grinding media. Note: Grinding media is not reused.

13. Store in the appropriate location for laboratory analysis.

Eurofins TestAmerica Canton's ISM Program Support

In 2003, the USACE recruited TestAmerica to join the ISM guidance development team at the Interstate Technology and Regulatory Council (ITRC). Dr. Mark Bruce was the primary TestAmerica representative on the team and continues his involvement to the present time. Dr. Bruce has provided ISM guidance to all of the network labs that provide ISM related services by standardizing procedures and ensuring that best practices are followed throughout the network.

The Canton lab has a dedicated drying room capable of holding 100- 200 samples at a time. The lab currently processes as many as 60 ISM samples a month. All processes are in compliance with ITRC guidance. The lab has been audited and approved in the past by several clients including state and federal agencies. The lab can process up to 18 ball mill samples a day. Over the last two years the lab has milled 173 samples.

Eurofins Test America believes that every ISM project starts with a site-specific plan that is best achieved with a pre-project conference call. Technical support is available to ensure that all ISM processing details and options are covered in in order to meet project requirements. General topics can include the following.

1. Air Drying: While most samples are air dried, there are instances where this may not be appropriate, depending on the contaminants of concern. The lab can process an ISM collected sample for one or more methods on an "as received" basis if the results might be impacted by air drying.
2. Sieving: The lab has a wide variety of sieve sizes that can be used and the discussion of project goals will help determine when and what constituents of a sample should go through the sieving process.
3. PSR (Particle size reduction): There are times when the particles remaining on the sieve are crushed and added back into the sample rather than being discarded.
4. Milling: The Canton lab has a ball mill whose capacity is 18-20 samples a day. The lab does not have a puck mill
5. Anticipated ISM sample size, QC needs, certification requirements and 2D slab cake details.

The Canton labs QA staff has reviewed St Louis's SOPs for ISM processing and ball milling. No significant differences were discovered.

Eurofins TestAmerica Denver

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Title: Acid Digestion of Aqueous Samples for Metals Analysis by ICP

Approvals (Signature/Date):

Doug Gomer — 10/6/20 Doug Gomer — Date Technical Manager	Reed Pottruff — 10/6/20 Reed Pottruff — Date Health & Safety Manager / Coordinator
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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the acid digestion of aqueous samples by EPA Method 200.7, SW-846 Method 3005A or SW-846 Method 3010A prior to the determination of the concentration of individual metallic elements by inductively coupled plasma atomic emission spectroscopy (ICP). These methods include digestions for total, total recoverable, dissolved, and potentially dissolved analytes (see definitions in Section 3).

1.2 This SOP is applicable to ground water, surface water, domestic and industrial wastewater, TCLP leachates, and other aqueous media. This SOP is not applicable to oils or other liquids that are not miscible with water.

NOTE: Samples that are found to be immiscible with water, e.g., contain oil or other immiscible organic solvents, are subcontracted to other labs that are capable of handling such samples. If during the preparation process it is discovered that the sample is immiscible with water or is biphasic, the analyst notifies the Technical Specialist and Project Manager, who can subcontract the samples to a laboratory with the capability to handle the sample.

1.3 The following table summarizes the applicability of the various digestion methods referenced in this SOP. All sample digestates are analyzed by ICP in accordance with SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

Method	Title	Summary	SOP Section
3005A/200.7_Prep	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP	Preparation of surface and ground water samples for total recoverable or dissolved metals for analysis by ICP.	10.5
3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP	Preparation of aqueous samples, EP and mobility procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.	10.8

1.4 Sample digestion requirements are established by the laboratory Project Manager before samples are received. Eurofins TestAmerica LIMS (TALS) method codes are applied to samples at Login to indicate which digestion is to be used for each sample.

1.5 This procedure can be used for all of the elements listed in Table 1. Additional elements may be analyzed using the digestion methods in this SOP provided the method performance criteria specified in Section 12 and the Quality Control (QC) acceptance criteria specified in Section 9 of this SOP and the ICP determinative SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021 are met.

1.6 All samples require digestion prior to analysis, with the possible exception of "direct analysis" of dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples. This must be determined by the laboratory Project Manager before projects start, and is communicated to the analysts through Method Comments in TALS.

2.0 Summary of Method

2.1 Method 3005A/200.7_Prep, Total Recoverable, Dissolved Metals or Potentially Dissolved Metals

A representative portion of sample is heated with diluted nitric and hydrochloric acids until substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2 Method 3010A Total Metals

A representative portion of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary), and brought up to volume.

3.0 Definitions

3.1 Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45-µm membrane filter prior to acidification (sample is acidified after filtration).

3.2 Potentially Dissolved Metals: The concentration of elements in solution after acidifying the sample with nitric acid to pH < 2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-µm membrane filter. This definition is based on the Colorado surface water regulations.

3.3 Total Recoverable Analyte: The concentration of analyte determined by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).

3.4 Total Metals: The concentration of elements in an unfiltered sample subject to a more rigorous nitric acid / hydrochloric acid digestion than is used for total recoverable metals.

3.5 General Analytical Terms: Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, “Quality Assurance Program,” for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work

areas, and atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not miscible with acids. If physical interferences are present, they should be documented in the final report case narrative.

4.3 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented in the final report case narrative.

4.4 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.5 Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Method 3005 or 3010 samples containing more than 1 mg/L silver are redigested at a reduced sample volume and reanalyzed to produce more accurate results. Method 200.7 requires samples to be redigested if the silver is greater than 0.1 mg/L.

4.6 Specific analytical interferences are discussed in the ICP determinative methods. See SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids are added.

5.3.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the digested samples.

5.4 Primary Materials Used

5.4.1 The following is a list of the materials used in this method which have a serious or significant hazard rating.

5.4.2 A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time.

Material (1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Stock Standard Solutions	Oxidizer Corrosive Poison	5 mg/m^3 as HNO_3	Toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid (HNO_3)	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (HCl)	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 Digestion blocks, with adjustable heating, capable of maintaining a sample temperature of 90 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C, in increments of 1 °C.

6.1.3 Liquid-filled thermometers must have a tag indicating that the accuracy was checked by the QA group within the last 12 months.

6.1.4 Digital thermometers must have a tag showing that they were checked within the last three months.

6.1.5 See SOP DV-QA-0001 for details of the thermometer calibration procedure.

6.1.6 Centrifuge (when the desired method of removing particulates is centrifugation).

6.1.7 Calibrated mechanical pipettes with disposable pipette tips. Pipette calibration is checked in accordance with SOP DV-QA-0008.

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters (No. 6973-2504), for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Re-pipettors or suitable reagent dispensers.

6.2.6 Class A volumetric graduated cylinders.

6.2.7 pH indicator strips.

6.2.8 Plastic digestate storage bottles.

7.0 Standards and Reagents

7.1 Standards must be NIST traceable, where available. Multi-element standards are verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), which is described in SOP DV-QA-0015.

7.2 Stock standards are purchased as custom multi-element mixes or as single-element solutions. Standards are logged into the TALS Reagent Module and are assigned unique identification numbers that can be used to access traceability information.

7.3 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles.

7.4 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.5 Standards containing silver must be protected from light using either a cardboard box or amber containers.

7.6 Shelf-Life

7.6.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.6.2 Intermediate concentration standards or working standards may be used for up to six months. The expiration date cannot be later than the date assigned to the stock standard.

7.6.3 Any suspect standards are re-verified, and replaced if re-verification fails.

7.7 Laboratory Control Sample (LCS) Spike Stock Standards

The LCS spike stock standards are custom-made standards purchased from CPI. The standards are designated TA-Spike1 (prep spike 1), TA-Spike 2 (prep spike 2), TA-Spike 3 (prep spike 3), TA-CM-OCT18-DEN-A and B (prep spike 4A and 4B) and SE Std Antimony (prep spike 5) and contain the following elements at ready-to-use concentrations:

LCS Spike Stock Standards

Elements in LCS Spike	Concentration in ppm (µg/mL)
As, Ba, Bi, Se, Si, Sn, Tl, U	200
B, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Sr, Th, Ti, V,	100

Elements in LCS Spike	Concentration in ppm (µg/mL)
Ca, K, Mg, Na	5000
Al, Fe, S	1000
P	2000
Zn, Zr	50
Ag	5

7.8 TCLP Spike Stock Standard (TCLP Spike)

The TCLP spike stock standard is purchased from commercial sources. The stock is a custom-made standard purchased at ready-to-use concentrations and designated as TCLP Spike, as follows:

TCLP Spike Stock Standard

Elements in TCLP Spike	Concentration in ppm (µg/mL)
Ba	1,000
Cr, Pb	500
As	300
Cu	200
Ag, Cd, Se	100

7.9 TCLP Mercury Spike Solution

TCLP leachate matrix spike samples are spiked for both ICP elements and mercury at the time of sample preparation but before preservation. The mercury spike standard is prepared by the mercury analyst as the mercury daily spike solution (Hg Daily Spk) at a concentration of 100 µg/L (SOP DV-MT-0015).

7.10 Reagent Water

Reagent water must be produced by a Millipore de-ionized system or equivalent and must achieve the performance specifications for ASTM Type II water, i.e., conductivity < 1.0 µmhos/cm; resistivity > 1.0 megohms-cm; silica < 3.0 µg/L. In addition, the reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

7.11 Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.12 Hydrochloric acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Eurofins TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD/DOE QSM 5.0 or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as

appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on initial demonstrations of capability, analyst training and qualification.

9.2 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.3 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are not included in the sample count unless specifically requested by the client. The prep batch consist of the laboratory generated QC and no more than twenty field samples.

9.4 Method Blank (MB)

9.4.1 The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples.

9.4.2 TCLP method blanks are prepared by taking 10 mL of TCLP leachate fluid (see SOP DV-IP-0012) through the appropriate procedure as described in Section 10. TCLP method blanks are referred to as LB (extraction fluid 1) and LB2 (extraction fluid 2) in TALS and on the final reports.

9.4.3 One method blank must be processed with each preparation batch. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of

elevated analyte concentrations or false-positive data. Method blank results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

9.4.4 Acceptance Criteria

The method blank should not contain any analyte of interest at or above ½ the reporting limit (RL) or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher. In other words, the sample result must be a minimum of 10 times higher than the blank contamination level. Method blank results that are greater than ½ the RL may also be reported if the associated sample results fall below the RL and the client accepts the data.

9.4.5 Corrective Action

If the method blank does not meet the acceptance criteria, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.5 Laboratory Control Sample (LCS)

9.5.1 One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples.

9.5.2 An LCS for a batch of aqueous samples is prepared by adding 0.5 mL of each of the spiking solutions listed in Section 7.7, to 50 mL of reagent water. This produces the final concentrations shown in Table 1.

9.5.3 An LCS for a TCLP batch is prepared by adding 0.5 mL of each of the LCS spike stock standards in Section 7.7, plus 0.5 mL of the TCLP Spike stock standard (Section 7.8) to 50 mL of the TCLP leachate solution (see SOP DV-IP-0012). This produces the final concentrations shown in Table 2.

9.5.4 The LCS is used to monitor the accuracy of the analytical process. LCS results are evaluated by the ICP analyst as described in SOP DV-MT-0012. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.5.5 Acceptance Criteria

LCS recovery control limits are set at ± 3 standard deviations about the historical mean. These limits must not be wider than 85 - 115 % recovery for Method 200.7 or 80 - 120 % for Method 6010. The control limits are maintained in TALS.

9.5.6 Corrective Action

If the LCS percent recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Normally, one MS/MSD pair is digested with each preparation batch. Samples identified as field blanks, equipment blanks, or rinse blanks are not appropriate for use as the batch MS/MSD.

9.6.2 Some programs (e.g., South Carolina and North Carolina) require that MS/MSD pairs are run at a 10% frequency. Also, some clients may require unspiked duplicate samples in place of or in addition to an MS/MSD pair. Check special project instructions attached as Method Comments in TALS and any project QASs before starting the batch.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, the LCS and LCSD are used to measure precision.

9.6.3 If insufficient sample is available to process an MS/MSD pair, then a duplicate LCS must be processed and an NCM generated. The LCS pair is then evaluated according to the MS/MSD criteria. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.6.4 The purpose of analyzing matrix spike samples is to assess the effect of the sample matrix on the accuracy and precision of the analysis. MS/MSD results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021. If the MS/MSD results fail to meet control limits while the LCS results are in control, then something about the sample matrix is interfering with the analysis.

9.6.5 Matrix spikes for aqueous sample batches are prepared by adding 0.5 mL of each of the LCS spike stock standards in Section 7.7, to a digestion tube containing 50 mL of the selected sample. The final spike concentrations are shown in Table 1.

9.6.6 Matrix spikes for TCLP batches are prepared by adding 0.5 mL of the TCLP Spike stock standard (Section 7.8) plus 0.5 mL of each of the LCS spike stock standards in Section 7.7, to 50 mL of the parent TCLP aliquot. A second aliquot is spiked for mercury analysis at by adding 1.5 mL of the 100 mg/L Hg standard (Hg Daily Spk) to 30ml of parent sample. The matrix spike samples are then preserved with HNO_3 to pH < 2. The final spike concentrations are shown in Table 2.

NOTE: The MS and MSD must be spiked prior to preservation of the leachate.

9.6.7 Acceptance Criteria

The recovery for each analyte must fall within established limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. If any analyte recovery or relative percent difference (RPD) between the MS and MSD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

9.6.8 Corrective Action

If MS/MSD results fail to meet control limits, but the LCS results are within limits, then samples do not require re-preparation and reanalysis unless the results indicate that a spiking error may have occurred. If the recovery of the LCS also failed acceptance criteria, then corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. One possible exception is an LCS recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

9.7 Continuing Calibration Verification Standard (CCV)

Continuing calibration verification standards (CCVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

9.8 Second-Source Initial Calibration Verification (ICV) Standard

Initial calibration verification standards (ICVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a non-conformance memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.

10.4 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.5 Sample Preparation

10.5.1 Samples are typically logged in as either water or solid. Waste such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), contact the project manager and the laboratory Technical Specialist for further instructions. It may be necessary to subcontract these samples to a laboratory with the capability to digest organic matrices.

NOTE: Eurofins TestAmerica Denver has not implemented digestion methods for water-immiscible organic matrices, e.g., oils. Samples that are known to be incompatible with Eurofins TestAmerica Denver digestion techniques are typically subcontracted to other laboratories.

10.5.2 All samples are to be electronically checked out of sample control using the TALS Internal Chain of Custody (ICOC) module.

10.5.3 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5.4 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through

the lab, data review and reporting.

10.5.5 Guidelines are provided in Appendix 1 on procedures to minimize contamination of samples and standards.

10.6 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure. The sample preparation procedures for Methods 3005A and 3010A detailed in the following sections are also summarized in work instruction WI-DV-016.

10.6.1 Verify sample pH

10.6.1.1 Measure the sample pH with pH paper using a separate aliquot of sample. This can be done using disposable plastic droppers or pouring the sample on to the pH paper. Do not put the pH paper directly into the bottle. Record the pH on a copy of the internal chain of custody (ICOC). When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

10.6.1.2 All water sample pH's must be verified and documented in the batch record before digestion.

10.6.1.3 If the pH>2 for a sample requiring acidic preservation, record the job in the Sample Filtration and Preservation Logbook.

10.6.1.4 If laboratory preservation is required, add 1-2 mL of conc. HNO_3 to the sample. Replace the lid and mix the sample. If the pH is still >2 add another addition of HNO_3. Do not add more than 5 mL. If the pH is still >2 create an NCM saying the sample will not preserve.

10.6.1.5 Allow the sample to sit for 24 hours following acidification.

10.6.1.6 Recheck the pH of the sample. If the pH>2, repeat Section 10.5.1.4 until the pH holds at <2 or 5 mL of HNO_3 has been added. If the pH is still >2 after the addition of 5 mL of HNO_3 create an NCM saying the sample will not preserve.

10.6.1.7 Samples cannot be digested for 24 hours after preservation. Note the date/time of this pH recheck in the Metals Prep Log in TALS.

10.6.1.8 Leachates or portions of leachates for metallic analyte deteminations must be acidified with nitric acid to a pH <2 unless precipitation occurs. Test a small portion of sample to see if precipitation occurs. If a precipitate forms do not acidify the leachate and analyze as soon a possible. Leachates may be digested as soon as they are acidified.

10.6.2 Select the unfiltered fraction for a total or total recoverable analysis or the filtered fraction for a dissolved analysis. If requested by the client, select the filtered fraction for a total dissolved analysis. For TCLP and SPLP, select the proper sample leachates.

10.6.2.1.1 Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number.

10.6.2.1.2 Samples and batch QC requiring filtration are to be put into a filtration batch. A filtration batch is to have no more than 20 samples.

10.6.2.1.3 Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples. The performance of the filtration process is recorded in TALS.

10.6.3 Mix the sample by shaking the container.

10.6.4 Measure and transfer 50 mL of the sample into a digestion tube (record the lot number of the digestion tubes used in TALS). When using calibrated digestion tubes, pour the sample into the tube to the 50 mL mark. For TCLP sample batches pour 10 mL of samples and bring to 50 mL with reagent water. Unless specifically required for a project, all samples are measured by volume and not by weight. Record the volume and units on the preparation bench sheet in TALS. If the digestion cup is filled beyond the required mark, the excess sample must not be poured back into the original container, but must be disposed of as waste.

10.6.5 Mix the sample by shaking the container and then measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot as described in Section 9.6. Refer to Section 9.6.6 for specific instructions for spiking the selected TCLP sample. Record the standards and pipette identifications in TALS.

10.6.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested (TALS 3005A), use filtered reagent water for the method blank. For TCLP sample batches, measure 10 mL of the TCLP leachate solution and bring to 50 mL with reagent water for the blank. See Section 9.4 for a detailed description of the method blank.

10.6.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add the spiking solutions as described in Section 9.6.2. For TCLP sample batches, use 10 mL of TCLP leachate fluid and bring to a final volume of 50 mL with reagent water for preparing the LCS (Section 9.5.3). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested and one or more samples were filtered in the laboratory, then filter the LCS using a filter of the same type that was used to filter the sample(s).

10.6.8 If the analysis is for total recoverable, dissolved metals, or potentially dissolved metals, continue on with Section 10.5. If the analysis is for total metals, skip Section 10.6 and go to Section 10.7.

10.7 Total Recoverable, Dissolved, or Potentially Dissolved Digestion for Waters by 3005A and 200.7_Prep.

10.7.1 Add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl to the sample in the digestion tube.

10.7.2 Heat at 90 - 95 °C until the volume is reduced to approximately 10 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.7.3 Allow the digestion tube to cool in a fume hood.

10.7.4 Wash down the digestion tube walls with reagent water.

10.7.5 Add 1.5 mL of concentrated HNO_3 to the digestate.

10.7.6 Revolume to 50 mL with reagent water. Cap and shake to mix.

10.7.7 If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.7.8 The sample is now ready for analysis.

10.8 Total Metals Digestion for Waters or TCLP Leachates by 3010A

10.8.1 Add 1.5 mL of concentrated HNO_3 to the sample in the digestion tube.

10.8.2 Heat at 90 - 95 °C until volume is reduced to approximately 5 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.8.3 Allow the digestion tube to cool in a fume hood.

10.8.4 Add another 1.5 mL portion of concentrated HNO_3 and cover the sample with a watchglass.

10.8.5 Continue refluxing until the digestion is complete.

NOTE: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient. Additional aliquots of nitric acid may be added if necessary.

10.8.6 Evaporate to a low volume of 5 to 10 mL. If the sample does go to dryness, the digestion must be started over using a fresh portion of sample.

10.8.7 Allow the digestion tube to cool in a fume hood.

10.8.8 Add 2.5 mL of concentrated HCl.

10.8.9 Cover and reflux for an additional 15 minutes to dissolve any precipitate or residue.

10.8.10 Wash down the digestion tube walls and watch glass (or digestion tube cover) with reagent water.

10.8.11 Adjust to 50 mL final volume with reagent water. This must be done volumetrically, and not using a balance.

10.8.12 If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

NOTES: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

10.8.13 The sample is now ready for analysis.

10.9 Calibration

10.9.1 The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded in TALS. The temperature must be monitored by measuring the temperature of reagent water contained in a capped digestion tube that is placed in each digestion block. The thermometer used and the start and end times for all temperature cycles are recorded in TALS.

10.9.2 The thermometer is calibrated in accordance with SOP DV-QA-0001, Thermometer Calibration Procedures.

11.0 Calculations / Data Reduction

11.1 This SOP does not produce any analytical data. See the determinative method SOPs DV-MT-0012, DV-MT-0019 or DV-MT-0021 for data analysis and applicable calculations.

11.2 Documentation

11.2.1 All of the preparation information is recorded and stored in TALS.

11.2.2 The preparation information includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample pH, Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number for each reagent used;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Start and stop times for digestions;

11.2.2.8 Observed and corrected temperature readings during digestion;

11.2.2.9 Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

11.3 Reporting

11.3.1 Reporting units are mg/L for water samples.

11.3.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.3.3 All associated data are entered or uploaded into TALS as required.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.4 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Eurofins TestAmerica SOP CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. MDL verifications are performed quarterly.

12.1.2 The current MDL values are maintained in TALS.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1 - 2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.2 Method 200.7, Determination of Metals And Trace Elements In Water And Wastes By Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, 1994.

16.0 Method Modifications

16.1 Modifications Specific to MCAWW Methods (200.7_Prep)

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 10. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness versus an exact volume).

16.2 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above ½ the reporting limit. Common laboratory contaminants are allowed up to the reporting limit in the blank following consultation with the client.

16.3 The referenced methods use 100 mL of sample for digestion. This SOP uses a 50 mL aliquot, with a proportional reduction in digestion reagents. This change is made to allow better control of temperature and potential sample contamination with the use of the digestion block. It is also considered one of the laboratory's hazardous waste reduction initiatives.

16.4 The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document states "flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1)

and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.0 Attachments

Table 1. Matrix Spike and Aqueous Laboratory Control Sample Levels

Table 2. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Appendix 1. Contamination Control Guidelines

18.0 Revision History

- Revision 13 dated 07 October 2020
  - Annual technical review

- Revision 12 dated 19 September 2019
  - Annual technical review
  - Updated spiking concentration information in Section 7.7
  - Updated spike concentrations in Tables 1 and 2

- Revision 11 dated 31 July 2018
  - Annual technical review
  - Updated Section 10.7.2 to reduce volume to approximately 10 mL
  - Updated Section 10.8.2 to reduce volume to approximately 5 mL

- Revision 10 dated 30 June 2017
  - Annual technical review
  - Updated Section 9.1.2 to include QSM 5.1
  - Added current Section 10.4 referencing support equipment IDs and renumbered remaining sections
  - Added Sections 11.3 and 11.4
  - Updated Section 12.1.1 MDLV language for consistency with other SOPs
  - Added current Section 12.2 regarding LOQVs and renumbered remaining sections
  - Updated the language in Sections 12.3 and 12.4 for consistency with other SOPs

- Revision 9 dated 30 June 2016
  - Annual technical review
  - Added "document in the batch record" regarding sample pH to Section 10.5.1.2.
  - Updated Section 9.5.2 to say 10ml of TCLP fluid is added to the method blank.
  - Updated Section 10.5.1.5 to wait 24 hours after acidification before checking pH
  - Converted the note in Section 10.5.2 into subsections 10.5.2.1.1 - 10.5.2.1.3
  - Added definition of filtration batch to section 10.5.2.1.2
  - Updated Section 16.2 to say we control common laboratory contaminates to the reporting limit

- Revision 8 dated 30 June 2015
  - Updated Section 10.5.1.1 to include statement about not putting the pH paper into the bottle
  - Added language to Section 4.5 for clarification
  - Added new Section 10.3 reminding analysts to enter data directly at time of

acquisition

- Revision 7 dated 31 October 2014
  - Annual technical review
  - Removed reference to SOP DV-IP-0017 for oils in section 1.2
  - Added maximum silver concentration to section 4.5 for method 200.7
  - Updated standard ID's for sections 7.7 and 7.8 and added Sulfur to the spike list
  - Corrected intermediate standard expirations from three months to six months
  - Removed duplicate analyte spike levels in ICP spike standards
  - Changed references from LIMS to TALS
  - Corrected concentration of Hg Daily spike standard
  - Removed Figures 1 and 2
  - Corrected various grammar and language errors
  - Corrected analyte spike levels in Table 1

- Revision 6 dated 08 October 2013
  - Updated sections 10.4.1.3, 10.4.1.4 and 10.4.1.6 about preservation procedure and removed the comment about recording the amount of acid added in the preservation logbook

- Revision 5, dated 15 July 2013
  - Annual review
  - Changed section 10.5.5, 7.3, 9.4, 9.5.2, 9.5.4, 10.3.1, 10.3.2, 10.4, 10.4.4, 10.5.2, 10.6.2, 11.2.2, 12.1.1 and 12.3 to reflect current practices
  - Corrected formatting and grammatical errors
  - Clarified sample matrices for this method in section 1.2
  - Corrected references in table associated with section 1.3
  - Added ICP determinative SOPs to sections 1.5, 4.6, 7.10, 9.5.3, 9.7.4
  - Added 200.7_Prep whenever 3005A was referenced
  - Edited section 3.5 to reflect current reference
  - Removed note associated with section 5.4.1
  - Added SOP reference to section 6.2.1
  - Removed references to Denver Standards Log and replaces those references with TALS reagent module
  - Correct standard names in section 7.7
  - Removed references to Supplemental Metals Prep Sheet
  - Updated sections 10.4.4, 10.4.6 and 10.4.7 for 10 mL TCLP sample aliquot
  - Added reference to 200.7 in Section 15

- Revision 4.7, dated 18 July 2012
  - Annual review
  - Updated Section 9.1, 10.1 and 10.2 to reflect current practice
  - Updated Section 9.7.6 on spiking TCLP aliquots
  - Added section 10.4.1.9 for TCLP preservation
  - Removed Appendix 2. Added reference to work instruction in Section 10.4
  - Updated Figures 1 and 2 to reflect current practice.
  - Formatting and editorial changes throughout

- Revision 4.6, dated 24 August 2011
  - Added recommendation to use disposable bulbs for pH checking in section 10.8.1.
  - Added requirement to store samples with a Rush form after preserving in section

10.8.1.2.

- Revision 4.5, dated 31 January 2011
  - Change note in section 10.8.1.8 to be 24 hours before preparation.

Earlier revision histories have been archived and are available upon request.

Table 1.

Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	LCS Concentration (µg/L)	Matrix Spike Concentration (µg/L)
Aluminum	10,000	10,000
Antimony	1,000	1,000
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	1,000	1,000
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	1,000	1,000
Calcium	50,000	50,000
Chromium	1,000	1,000
Cobalt	1,000	1,000
Copper	1,000	1,000
Iron	10,000	10,000
Lead	1,000	1,000
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	1,000	1,000
Molybdenum	1,000	1,000
Nickel	1,000	1,000
Phosphorous	20,000	20,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	2,000	2,000
Si (as SiO2)	4.28	41,028
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Thallium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	1,000	1,000
Zinc	500	500
Zirconium	500	500

Table 2.

TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Element	RL (mg/L)	Regulatory Limit (mg/L)	Spike Level (mg/L)
Arsenic	0.1	5,000	5.0
Barium	1.0	100,000	12.0
Cadmium	0.05	1,000	2.0
Chromium	1.0	5,000	6.0
Lead	0.03	5,000	6.0
Selenium	0.05	1,000	3.0
Silver	0.1	5,000	1.05

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Appendix 1.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: Acid Digestion of Aqueous Samples for Analysis by ICP-MS

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) using EPA Method 200.8, and SW-846 Methods 3005A and 3020A.

1.2 Aqueous samples also include TCLP & SPLP Leachates, aqueous equipment rinse blanks for soil sampling. In some cases, where the associated soil samples require the SW-846 Method 3050B, Section 7.5, optional treatment to improve solubility and recovery of Sb, Ag, and Sn, the client may require that the aqueous equipment blank receive the same treatment. Refer to section 10.14 for this prep.

1.3 The applicability of each of these preparation protocols to specific analytes is detailed in the TestAmerica LIMS System (TALS). Additional elements may be analyzed following digestion by these protocols, provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.4 This SOP provides procedures applicable to the preparation of dissolved, total recoverable, potentially dissolved, and total metallic elements in ground water, aqueous samples, aqueous sludges, aqueous wastes, aqueous air sampling media, and leachates/extracts. This SOP is not applicable to samples that contain or consist of oil or other immiscible organic solvents.

NOTE: Samples that are known to be immiscible with water, e.g., contain or consist of oil or other immiscible organic solvents should be logged with a waste matrix and subbed out to a different TestAmerica laboratory for digestion and analysis.

1.5 SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP-MS. Although digestion is not specifically required by the method (SW-846 3005A section 2.2) for dissolved samples, the standard operating procedure at TestAmerica Denver is for all matrices to be digested prior to analysis.

1.6 EPA Method 200.8 Section 11.2 is used to prepare surface water, drinking water, and domestic and industrial waste samples for total recoverable and dissolved metals.

1.7 SW-846 Method 3020A is used to prepare aqueous samples, TCLP leachates, SPLP leachates and aqueous wastes that contain suspended solids for total metals analysis by ICP-MS.

1.8 The following table lists the sample preparation methods that are covered in this SOP and the specific section of this SOP for each preparation method. Prepared samples are analyzed by inductively coupled plasma-mass spectrometry (ICP-MS).

PREPARATION METHOD	SOP SECTION	DETERMINATIVE METHOD	ANALYTICAL SOPS #
Method – 3020A Total	10.11	ICP-MS	DV-MT-0018 DV-MT-0022
Method – 3005A Total Rec./Dissolved	10.12	ICP-MS	DV-MT-0018 DV-MT-0022
Method 200.8 – Total Rec.	10.13	ICP-MS	DV-MT-0002
Method 200.8 – Dissolved	10.13	ICP-MS	DV-MT-0002
Method - 3050B Special Sb Prep	10.14	ICP-MS	DV-MT-0018 DV-MT-0022

2.0 Summary of Method

2.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals.

This preparation method is used for total recoverable and dissolved metals analysis by ICP-MS method 6020 and 6020A. A representative aliquot of sample is heated with nitric acid and substantially reduced in volume. The digestate is diluted to volume and then filtered (if necessary).

2.2 Method 3020A, Acid Digestion of Aqueous Samples and TCLP/SPLP Leachates for Total Metals.

This preparation method is used for total metals analysis by ICP-MS method 6020 and 6020A. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. The digestate is diluted to volume and then filtered (if necessary).

2.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry.

This preparation method is used for metals analysis by ICP-MS method 200.8. A representative aliquot of sample is refluxed with nitric and hydrochloric acids. The digestate is diluted to volume and then filtered (if necessary).

3.0 Definitions

Additional definitions of terms used in this SOP may be found in the glossary of the QAM.

- Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45-μm membrane (Method 3005A). (The sample is acidified after filtration).

- Total Metals: The concentration of metals determined in an unfiltered sample following digestion (Method 3020A).

- Total Recoverable Metals: The concentration of metals determined in an unfiltered sample following treatment with hot, dilute mineral acid (Method 200.8 and Method 3005A).

- Potentially Dissolved Metals: An acidified sample is filtered between 8- 96 hours following acidification and the filtrate is digested using Method 3005A.

- Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include the following: metallic or metal-containing lab ware (e.g., latex gloves coated with talc, which contains high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.4 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.5 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared.

4.6 Specific analytical interferences are discussed in the ICP-MS determinative method SOPs, e.g., DV-MT-0002, DV-MT-0018, and DV-MT-0022.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns

5.1.1 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids

are added.

5.1.2 The digestion solution must be cooled sufficiently before adding hydrogen peroxide (H_2O_2) to avoid a reaction and possible violent effervescence, or boiling over of the digestion solution.

5.1.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the sample digestate.

5.1.4 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	**Hazards**	**Exposure Limit** [(2)]	**Signs and Symptoms of Exposure**
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm (TWA)	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion block, with adjustable heating, capable of maintaining a sample temperature of 85 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C.

6.1.3 Centrifugation equipment (if desired method of removing particulate material is centrifugation).

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Repipettors or suitable reagent dispensers.

6.2.6 Calibrated automatic pipettes with pipette tips or Class A glass volumetric pipettes.

6.2.7 Class A volumetric flasks.

6.2.8 pH indicator strips (pH range 0 - 6).

6.2.9 Plastic digestate storage bottles.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative method SOPs, e.g., DV-MT-0002, DV-MT-0018, and DV-MT-0022.

7.2 Laboratory control sample (LCS), and matrix spike and matrix spike duplicate (MS/MSD) spike solutions are purchased as custom TestAmerica Denver solutions. Standards are logged into the Reagents module of TALS and are assigned unique identification numbers that can be used to access traceability information. The TALS identification numbers are recorded on the metals prep bench sheet.

7.2.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. These plastic bottles may be stored in a glass jar.

7.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3 See TALS for the list of spiking levels. A volume of 0.1 mL of each working spike solution is added to the 50-mL final sample volume.

7.3 Nitric Acid (HNO_3), concentrated, trace-metal grade or better.

NOTE: When preparing diluted acids, always add acid to water. If the water is added to the acid, the sudden increase in temperature may cause splashing.

7.4 Nitric Acid, 1:1

Dilute concentrated HNO_3 with an equal volume of reagent water.

7.5 30% Hydrogen Peroxide (H_2O_2), ultra pure grade.

7.6 Hydrochloric Acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE Or Glass	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS login, sample or method comments and/or program QAS to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified appropriately. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Table 2 provides a summary of quality control requirements, including type, frequency, acceptance criteria, and corrective action. Detailed information regarding acceptance criteria and corrective action are found in each determinative method SOP.

9.3 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See

Section 12.1 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.4 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). For samples logged in under Method 200.8, there must be two MS/MSD pairs for every batch containing more than ten samples. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

9.5 Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are usually not included in the sample count.

9.6 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.7 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs)

may require the use of sample duplicates in place of or in addition to MS/MSDs. At least one MS/MSD pair must be processed for each preparation batch. Some client programs require a 10 % MS/MSD analysis frequency. If insufficient sample is available to process an MS/MSD pair, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QASs) are developed to address these requirements.

10.0 Procedure

Sample Preparation

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervisors to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3 All samples are to be checked out of Sample Control with the Internal Chain of Custody in TALS properly completed.

10.4 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5 Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be more like a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), then contact the project manager and the laboratory group leader for further instructions.

10.6 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review, and reporting groups.

10.7 Guidelines are provided in Appendix A on procedures to minimize contamination of samples and standards.

10.8 All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.

10.9 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure:

10.9.1 Sample pH is verified during sample receipt. When a sample is received with improper/insufficient preservation, the sample is delivered with notification of the deficiency.

10.9.1.1 Measure the sample pH with pH paper using a separate aliquot of sample. Do not put the pH paper directly into to bottle. Record the pH on a copy of the internal chain of custody (ICOC). When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

10.9.1.2 If the pH>2 for a sample requiring acidic preservation, add 1-2 mL of conc. HNO_3 to the sample. Replace the lid and mix.

10.9.1.3 Recheck the pH of the sample. If the pH<2, record the sample in the Metals preservation logbook. If the pH>2, repeat 10.9.1.2 until pH<2 or 5mls has been added. If the sample still has a pH greater than 2 do not add any additional acid and create an NCM. Add the sample to the Metals preservation logbook

10.9.1.4 Allow the sample to sit for 24 hours following acidification.

10.9.1.5 After 8-16 hours, recheck the pH of the sample. If the pH<2, proceed with the appropriate digestion procedure. Note the date/time of this pH recheck in the Metals preservation logbook.

10.9.1.6 If after 8-16 hours the pH>2, repeat steps 10.9.1.2 through 10.9.1.5 until the pH remains <2 following the 8-16 hour period or 5mls of HNO_3 has been added.

Note: Acid must be added at least 24 hours before analysis.

10.9.2 Select the unfiltered fraction for a total or total recoverable analysis or the

filtered fraction for a dissolved analysis. For SPLP select the proper sample leachates.

NOTE: Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number. Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples.

10.9.3 Mix the sample by shaking the container.

10.9.4 Measure and transfer 50 mL of the sample into a digestion tube. When using calibrated digestion tubes, pour the sample into the tube to the 50-mL mark. Record the lot number of the digestion tubes in TALS.

10.9.5 Measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot with 0.1 mL of each spiking solution (see TALS). Record the standards and pipette identifications in TALS.

10.9.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested, use filtered reagent water for the method blank.

10.9.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add 0.1 mL of spiking solution (see Table 2). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested (preparation method 3005A), and one or more samples were filtered in the laboratory, then filter the LCS and Method Blank using a filter of the same type that was used to filter the sample(s).

10.10 Proceed to the appropriate Section of this SOP for the desired preparation method as follows:

Preparation Method*	SOP Section	Analytical Method
3020A Total Metals	10.11	Method 6020
3005A Total Recoverable	10.12	Method 6020
3005A Dissolved Metals	10.12	Method 6020
200.8 Total Recoverable Metals	10.13	Method 200.8

200.8 Dissolved Metals	10.13	Method 200.8
3050B Special Sb prep	10.14	Method 6020

(See also WI-DV-017)

10.11 Method 3020A - Preparation for Total Metals Analysis by ICP-MS Method 6020 and 6020A

10.11.1 To the sample in a digestion tube, add 1.5 mL of concentrated HNO_3.

10.11.2 Heat at 90 - 95 °C until the volume is reduced to approximately 5 mL. Record the start time and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared and reanalyzed.

10.11.3 Allow the digestion tube to cool in a fume hood.

10.11.4 Add 1.5 mL of concentrated HNO_3. Cover and reflux gently.

10.11.5 Continue heating, adding additional acid as necessary in 1-2 mL increments to ensure a complete digestion. Record the start and stop times and the Hot Block temperature in TALS.

NOTE: Digestion is complete when the digestate is light in color and does not change in appearance with continued refluxing.

10.11.6 Evaporate to low volume, approximately 3 - 5 mL.

10.11.7 Allow the digestion tube to cool, then add about 10 mL of reagent water.

10.11.8 Replace the cover and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur. Record the stop time in TALS.

10.11.9 Allow the sample to cool and rinse the watch glass into the digestion tube with reagent water.

10.11.10 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.11.11 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018 or DV-MT-0022.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 or DV-MT-0022 for additional details.

10.12 Method 3005A - Preparation for Total Recoverable and Dissolved Metals Analysis by ICP-MS Method 6020 and 6020A

10.12.1 To the sample in a digestion tube, add 2.0 mL of concentrated HNO_3.

10.12.2 Heat the sample to 90 - 95 °C and cautiously evaporate to approximately 10 mL, while ensuring that no portion of the sample container is allowed to go dry. Record the start and stop times and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.12.3 Allow the sample to cool in a fume hood.

10.12.4 Rinse the digestion tube with reagent water.

10.12.5 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.12.6 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0018 or DV-MT-0022 for additional details.

10.13 Method 200.8 - Preparation for Total Recoverable/Potentially Dissolved/Dissolved Metals Analysis by ICP-MS

10.13.1 To the sample, add 0.5 mL of concentrated HNO_3 and 0.25 mL of concentrated HCl.

10.13.2 Adjust the digestion block temperature so the solution in a covered container rises to approximately 90 - 95 ºC. Record temperature on bench sheet.

10.13.3 Heat the sample until it evaporates to approximately 10 mL, while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.13.4 Cover the sample and gently reflux for an additional 30 minutes. Avoid vigorous boiling to prevent the loss of the HCl-H_2O azeotrope. Record the start and stop times and the Hot Block temperature in TALS.

10.13.5 Allow the sample to cool in a fume hood.

10.13.6 Rinse the watch glass or cover into the container and re-volume to 25 mL with reagent water. Cap and mix thoroughly.

NOTE: If the samples are being prepared to satisfy drinking water compliance requirements, bring the samples to a final volume of 50 mL before capping and mixing. Samples logged for this purpose will have attached TALS Method Comments to that effect.

10.13.7 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0002.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0002 for additional details.

10.14 Method 3050B – Special Prep for Sb, Sn and Ag for Analysis by ICP-MS Method 6020

10.14.1 To 25 mL of sample in a digestion tube, add 2.5 mL of HNO_3 and 2.5 mL of HCl.

10.14.2 Heat at 90-95 °C until the sample has reduced to a volume of 10-15 mL ensuring that no portion of the sample container is allowed to go dry.

Record the start and stop times and the Hot Block temperature in TALS.

10.14.3 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.14.4 Add 1.0 mL of HCl to the digestion tube and cover with a ribbed watch glass.

10.14.5 Replace the watch glass and heat the sample for 15 minutes.

Record the start and stop times and the Hot Block temperature in TALS.

10.14.6 Remove the sample from the Hot Block and allow it to cool in a fume hood.

10.14.7 Re-volume to 100 mL with reagent water, cap and mix thoroughly.

10.15 Calibration

The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded on the metals preparation bench sheet. The temperature must be monitored by measuring the temperature of reagent water contained in a digestion tube that is placed in each digestion block. The thermometer used and the start and end time temperatures are recorded in TALS. The thermometer is calibrated in accordance

with SOP DV-QA-0001, *Thermometer Calibration Procedures*.

11.0 Calculations / Data Reduction

11.1 Not applicable. See the determinative method SOPs, DV-MT-0002, DV-MT-0018 and DV-MT-0022 for data analysis and applicable calculations.

11.2 Documentation

11.2.1 All of the preparation information is recorded and stored in TALS.

11.2.2 The preparation information includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample pH, Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number for each reagent used;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Start and stop times for digestions;

11.2.2.8 Observed and corrected temperature readings during digestion;

11.2.2.9 Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.3 Method 6020, Inductively Coupled Plasma - Mass Spectrometry, Revision 0, September 1994.

15.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

15.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectroscopy, Revision 5.4, May 1994.

16.0 Method Modifications:

16.1 Modifications and Interpretations Applicable to SW-846 Reference Methods

16.1.1 Chapter 1 of SW-846 states that the method blanks should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above one-half of the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.1.2 The referenced methods, as well as Table 3-1 of SW-846, refer to the use of a 100-mL aliquot for digestion. This SOP requires the use of a 50-mL sample size to reduce waste generation. The use of reduced sample volumes is supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition", dated November 3, 1994. This document stated, "...flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..."

EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a

significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated. " As a 'representative sample' can be assured, scaling causes no loss of precision and accuracy in the analysis."

16.2 Modifications Specific to Method 3005A

An additional 1.0 mL of HNO_3 was included to replace the 5.0 mL of HCl. HCl was eliminated to reduce interferences from chloride.

16.3 Modifications and Interpretations Specific to Method 3020A

16.3.1 Section 10.11.6 of this SOP requires that the sample be reduced to a volume of 3-5 mL. Section 7.2 of Method 3020A states that the volume should be reduced to 3 mL, but also states that no portion of the bottom of the digestion tube should go dry. The volume required by this SOP is a closer approximation of the volume required to provide an adequate covering of the bottom of the digestion tube so as to prevent the loss of critical analytes through volatilization.

16.3.2 The scope of 3020A has been expanded to include silver, based on comparison studies with 7760A. Method 3020A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water, and TCLP leachate) up to a concentration of 1 ppm silver.

17.0 Attachments

Table 1. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Table 2. Summary of Quality Control Requirements
Appendix A. Contamination Control Guidelines

18.0 Revision History

- Revision 12, Dated 31 October 2019
  - Annual Review
  - Update spiking levels in Table #1

- Revision 11, Dated 31 October 2018
  - Annual Review
  - Updated Section 10.12.2 volume to approximately 10 mL.
  - Updated spike amount in sections 10.9.5 and 10.9.7.

- Revision 10, Dated 31 October 2017
  - Annual Review
  - Removed Note about sample count for AFCEE

- Revision 9, Dated 30 September 2016
  - Annual Review
  - Removed reference to Method 3050 from title page and Sections 1.1 and 15.
  - Removed Acceptance Criteria and Corrective Actions for QC samples from Section 9. These are addressed in each determinative SOP.
  - Updated Section 10.9.1.4 preservation time to 24 hours

- Added method blank to section for filtration if samples are filtered. All QC must be treated the same.
- Revised Section 12 to reflect current practice
- Revised Table 2 to include all determinative method SOPs

- Revision 8, Dated 30 September 2015
  - Added drinking water to Section 1.6.
  - Corrected Section reference tables.
  - Added note to Section 10.13.6 describing changed final volume requirement.

- Revision 7, Dated 30 June 2015
  - Updated Section 10.13.2 for temperature requirement of 90-95 for method 200.8
  - Updated Section 10.9.1.1 for proper technique when using pH paper.
  - Added new Section 10.8 reminding analysts to enter data directly at time of acquisition
  - Added Section 11.2 describing required data to be recorded
  - Removed Section 11.4

- Revision 6, Dated 29 September 2014
  - Annual review
  - Removed references to microwave procedure
  - Removed direct-shoot for dissolved analysis
  - Corrected section references
  - Replaced LIMS and Standards Log references with TALS
  - Clarified the number of MS/MSDs per prep batch based on method
  - Removed workflow diagrams Figures 1-4
  - Removed Tables 1 and 2
  - Minor spelling and language corrections throughout

- Revision 5, Dated 30 September 2013
  - Reference to SOP DV-IP-0017 for preparation of organic waste
  - Formatting updates
  - Updated section 9, 12 & 14 to include more detail
  - Annual review
  - Updated sections 10.8.1.2 - 10.8.1.4 to removed reference to amount of HNO_3 acid added.
  - Added to Section 10.8.1.6 that a maximum of 5mls of HNO_3 can be added.

- Revision 4, Dated 28 September 2012
  - Annual review
  - Section 9.6 Updated method blank control limits to ½ the reporting limit.
  - Updated appendix B with revised Work Instruction.

- Revision 3.5, dated 23 September 2011
  - Annual Technical Review
  - Removed reference to Supplemental Metals Prep Sheets in Sections 10.10.8 and 10.13.5.1
  - Removed reference to Clouseau in Section 10.8.1.2
  - Removed references to LIMS codes in Appendix B

Earlier revision histories have been archived and are available upon request.

Table 1.

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	3200
Barium	10000	100000	10200
Cadmium	100	1000	1200
Chromium	500	5000	5200
Lead	500	5000	5200
Selenium	250	1000	1200
Silver	500	5000	1200

Table 2.

Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-prep not required unless preparation error suspected.
Matrix Spike Duplicate (MSD)	See Matrix Spike frequency	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	See Corrective Action for Matrix Spike

Appendix A.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: ACID DIGESTION OF SOLIDS [Method EPA 3050B]

Approvals (Signature/Date):

Doug Gomer 3/2/20 Doug Gomer — Date Technical Specialist	Reed Pottruff 3/2/2020 Reed Pottruff — Date Health & Safety Manager / Coordinator
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1.0 Scope and Application

1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).

1.2 Method 3050B is designed to determine the concentration of "environmentally available" metals, and is not a true "total metals" digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.

1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by TestAmerica Denver is shown in Table II.

1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

2.0 Summary of Method

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10 g of sample is used and brought to a final volume of 500 ml.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.2 **Total Metals -** Although Method 3050B is often referred to as a "total metals" digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, "This method is not a total digestion technique for most samples." There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.

3.3 **Preparation Batch -** A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.

3.4 **Reagent Water –** Water that is free of the analytes of interest. In the Metals group, reagent water is obtained from a Barnstead E-Pure water purification system.

3.5 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*.

4.0 Interferences

4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.

4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.

4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP-MS procedure that includes HCl as the final digestion acid. See Section 11.12 of this SOP.

4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. See Attachment 1 for more information regarding contaminant control.

4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.6 For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.

4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not

behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.

4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid. Increasing the volume of hydrochloric acid can produce results approaching the true copper concentration. Samples that appear to have nonstandard matrices or have visible metal particles should be documented in an NCM.

4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.

4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic materials or samples that are at elevated pH can react violently when acids are added. If any solid sample appears to be a chemical substance rather than an environmental sample, consult with the group supervisor or the Project Manager (PM) before adding acid.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and

reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Hydrogen Peroxide, H_2O_2	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m^3 TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid, HCl	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.

(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

Note: Balances are serviced annually and the accuracy checked daily using three standard masses. See SOP DV-QA-0014 for details.

6.1.2 Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95 °C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is partially filled with water. The water in the tube must be high enough to cover the thermometer past the minimum immersion line. The temperature is directly recorded in the Batch Information area in the TestAmerica LIMS (TALS).

6.2 Supplies

6.2.1 Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110 °C with clearly visible 1 °C increments.

Note: Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001.

6.2.2 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.3 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.4 Whatman 541 (acid washed) filter paper, or equivalent.

6.2.5 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.6 Syringes or equivalent filtration apparatus.

6.2.7 Disposable plastic funnels.

6.2.8 Disposable wooden spatulas for subsampling.

6.2.9 Centrifuge, capable of at least 2,000 rpm.

6.2.10 Graduated cylinders, 100 mL and 500 mL, capable of ± 3% accuracy.

6.2.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

Note: Mechanical pipettes are calibrated before use as described in SOP DV-QA-0008.

6.2.12 Class A volumetric flasks.

6.2.13 pH indicator strips (pH range 0 – 6).

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.

7.2 Nitric acid (HNO_3), concentrated - Trace metal grade or better.

7.3 Nitric acid (HNO_3), 5% - Add 50 mL of concentrated HNO_3 to approximately 900 mL of reagent water and dilute to 1 liter.

7.4 Hydrochloric acid (HCl), concentrated - Trace metal grade or better.

7.5 30% Hydrogen peroxide (H_2O_2) - Reagent grade used for ICP analysis.

7.6 30% Hydrogen peroxide (H_2O_2) – Ultra pure used for ICP-MS analysis.

7.7 Glass beads, ≤ 1 mm diameter, washed with aqua regia (for DoD projects).

7.8 Standards

7.8.1 All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before the initial use, as described in SOP DV-QA-0015.

7.8.2 Storage and Shelf Life of Metal Standards

7.8.2.1 Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

7.8.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.8.3 LCS and MS Spike Solutions

7.8.3.1 ICP and ICP/MS spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed.

7.8.3.2 If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

7.8.3.3 Intermediate standards prepared in the laboratory may be used for spiking as long as the procedures for standard recording and verification outlined in SOP DV-QA-0015 are followed.

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix #1	As, Se, Si, Sn, Tl Ba, Be, Cd, Co, Cr, Li, Mn, Mo, Ni, Pb, Sr, Ti, V	200 100
ICP	Spike Mix #2	Ca, K, Mg, Na Al, Fe	5000 1000
ICP	Spike Mix # 3	P S U W	2000 1000 200 100
ICP	Spike Mix # 4	Bi B, Th Zn, Zr	200 100 50

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix # 4B	Ag	5
ICP	Spike Mix # 5	Sb	100
ICP-MS	MS CALSTD-1	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn Sr, Ti, Tl, V	100
ICP-MS	ICP CALSTD-2	Al, Ca, Fe, K, Mg, Na	2000
ICP-MS	MS Spike 2	Ag, Sr, Th, U, W, Zn	20

Note: ICP or ICP-MS digestions may select different combinations of spikes in order to satisfy client requests. All spikes used for sample digestion will be recorded in the Reagent module in TALS.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at ≤6 °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Soils	Glass	3 grams	Cool ≤ 6 °C	180 Days	N/A

[1] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

[2] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation batches may consist of up to 20 field samples. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.3 Minimum QC Requirements

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in TALS. If clients specify specific samples for the MS and MSD, the batch may contain multiple MS/MSD pairs.

9.3.1 Method Blank (MB)

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11. Add 1.0 g of prewashed glass beads to the blank if required by the client to better simulate a solid matrix.

The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

Corrective Action: If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.3.2 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS contains reagent water that is spiked with all the analytes of interest and is carried through the entire analytical procedure. A duplicate LCS (LCSD) must be prepared when there is insufficient sample volume to perform an MS/MSD. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Add 1.0 g of prewashed glass beads to the LCS if required by the client to better simulate a solid matrix.

The spike solutions described in Section 7.8.3 are used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- Routine ICP-MS: Add 1.0 mL of spike
- DoD ICP-MS: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table 2 and Table 3.

Incremental Sampling Method LCSs are spiked with 5 ml of spike.

Acceptance Criteria: Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

Corrective Action: When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP-MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Tables II through IV. Incremental Sampling Method MS/MSD pairs are spiked with 5 ml of spike.

NOTE 1: The spike must be added after the sample aliquot but before any reagents.

NOTE 2: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD pair is not available, an LCS and LCSD are used to measure precision.

10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed

must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Custody

11.3.1 Samples are transferred from the Sample Control group to the Metals group and the transfer is documented using the Sample Transfer function of the Internal Chain of Custody in TALS (see SOP DV-QA-0003 for details).

11.3.2 Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

11.4 Subsampling

11.4.1 It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples must be mixed and incrementally subsampled to obtain a representative portion. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023, *Subsampling*, for directions.

11.4.2 Select at least three incremental subsamples from different locations in the original sample and place them in a tared 50 mL digestion tube. The final sample weight should be between 1.0 and 1.5 g. Record the weight to the nearest 0.01 g.

11.4.3 Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

NOTE: When adding glass beads to the Method Blank and LCS digestion tubes, the nominal weight must be entered into the Initial Amount field in TALS. The true weight of glass beads should be recorded in the Notes field on the Worksheet tab in the preparation batch.

11.5 Incremental Sampling Method Digestion

For the Incremental Sampling Method approximately 10 g of sample is weighed out by the Organic Prep group following the procedure described in SOP DV-OP-0013. This pre-weighed sample is then delivered to the Metals group for digestion and analysis. The sample weight is recorded on the ISM Worksheet and attached to the incremental sampling batch in TALS. The pre-weighed aliquots are delivered in 125 mL digestion tubes which are ready for spike standards and reagents to be added. The Method 3050B digestion reagents are increased 5x to maintain the same proportions as are used for a 1-2 gram sample. When required, 10 g of glass beads are added to the Method Blank and LCS prior to digestion.

11.6 Initial Digestion Cycle with 1:1 Nitric Acid

11.6.1 Add approximately 5 mL of reagent water to each digestion tube.

11.6.2 Add 5 mL of concentrated HNO_3.

11.6.3 After all of the acid has been added to the preparation batch, gently swirl the samples to mix and then place the sample rack on the Hot Block.

11.6.4 Place a ribbed cover on each tube.

11.6.5 Heat samples to 90-95 °C, and reflux for 15 minutes without boiling.

NOTE: DO NOT ALLOW SAMPLES TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.6.6 Remove the samples from the Hot Block and allow them to cool before proceeding with the next step.

11.6.7 Record the start time, starting temperature, end time, and ending temperature in TALS.

11.7 Second Digestion Cycle Using Concentrated Nitric Acid

11.7.1 Add 5 mL of concentrated HNO_3, and replace the ribbed cover.

11.7.2 Place samples back on the Hot Block and reflux at 90-95 °C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.7.3 If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO_3 acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

11.7.4 Heat the samples at 90-95 °C for 2 hours.

11.7.5 Allow the samples to thoroughly cool before proceeding.

11.8 Third Digestion Cycle Using Hydrogen Peroxide

11.8.1 Add 2 mL of reagent water to each tube.

11.8.2 Add 3 mL of 30% H_2O_2 a few drops at a time. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.8.3 Replace the ribbed cover and heat samples until effervescence subsides.

11.8.4 Allow the samples to cool.

11.8.5 Continue adding 30% H_2O_2 in 1 mL increments with warming until effervescence is minimal or sample appearance is unchanged. If additional peroxide is added to a sample then it must also be added to the method blank and LCS.

NOTE: Do not add more than a total of 10 mL of 30% H_2O_2. If 10 mL have been added and the samples are still vigorously effervescing, document the situation with an NCM and continue with the digestion.

11.8.6 Heat the samples at 90-95 °C for 2 hours.

11.8.7 Allow the samples to cool.

11.8.8 If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in Section 11.8. If the samples will be analyzed by ICP-MS, skip the HCl digestion step and go to step 11.10.

11.9 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid

11.9.1 If the samples are being prepared for ICP analysis, add 10 mL of concentrated HCl to the samples in the digestion tubes and cover with ribbed covers.

11.9.2 Reflux for an additional 15 minutes without boiling.

11.9.3 Allow the samples to cool.

11.10 Separating Undigested Solids from the Digestion Solution

11.10.1 Filter samples through Whatman 541 or equivalent fiber filters into a graduated 125 mL digestion tube whose accuracy is documented to be better than ± 3%.

NOTE: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.2 For samples digested by the Incremental Sampling Method use a 500 mL poly bottle that has been measured after measuring out 500 mL of DI water from a graduated cylinder.

11.10.3 Wash the original digestion tube and ribbed cover with reagent water to ensure quantitative transfer of all of the digestion solution into the new digestion tube.

11.10.4 Rinse the funnel and filter paper with reagent water to ensure complete sample transfer into the new digestion tube.

11.10.5 Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TALS.

For Multi-Incremental samples the final volume is 500 mL.

11.11 Documentation and Record Management

The following information must be recorded for each preparation batch. This information is directly entered into TALS.

- Initial sample weight and final digestion volume
- Preparation analyst and date
- Identification of all reagents and standards
- Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes)
- Glass beads lot number
- Filter paper lot number
- Digestion tube lot number
- Hot Block ID number
- Fume Hood ID number

11.12 Alternate Antimony Preparation for Analysis by ICP-MS

11.12.1 Weigh out 1.0-1.5 g soil samples according to the procedure in Section 11.3.

11.12.2 Add approximately 5 mL of reagent water to each digestion tube.

11.12.3 Spike the LCS, LCSD, MS, and MSD with 1.0 mL of the MS spike 2 standard.

11.12.4 Add 2.5 mL concentrated HNO_3 and 2.5 mL concentrated HCI to each sample and batch QC.

11.12.5 Cover each tube with a watch glass and reflux on hot block at 90-95 °C for 15 minutes.

11.12.6 Filter through Whatman 541 or equivalent filter paper into a new 125 mL digestion tube while still hot.

11.12.7 Rinse the filter and funnel with 1.25 ml of hot (~95 °C) concentrated HCI.

11.12.8 Rinse three times with hot (~95 °C) reagent water (5 mL rinses.)

11.12.9 Place the filter paper and soil residue back into the original sample

digestion vessel. Add 2.5 mL concentrated HCl, cover and reflux on the hot block for 20 minutes or until paper dissolves.

11.12.10 Filter through a fresh filter into the original filtrate. Rinse three times with reagent water (5 mL rinses).

11.12.11 Bring to final volume of 100 mL with reagent water.

12.0 Calculations / Data Reduction

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.3 Training Requirements

The group leader or supervisor is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the

required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered in the batch record (View Batch Information)until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J

15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

17.0 Method Modifications:

Item	Method	Modification

1	3050B	Method 3050B Section 7.1 states that a 1-2 g aliquot is to be used. The amount specified by TestAmerica Denver in this procedure is limited to 1-1.5 g in order to prevent increased instrument maintenance and sample reruns due to dilutions.

18.0 Figures, Tables, and Attachments

Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS

Table 2: Soil LCS and MS/MSD Spikes for ICP

Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines

19.0 Revision History

- Revision 14 dated 3 March 2020
  - Updated copyright information
- Revision 13 dated 31 January 2020
  - Annual Review
  - Updated spiking standard concentrations in Sections 7.8.3.3, Tables 2 and 3
- Revision 12 dated 31 October 2019
  - Annual Review
- Revision 11 dated 31 October 2018
  - Annual Review
  - Updated copyright information
- Revision 10 dated 31 October 2017
  - Annual Review
- Revision 9 dated 31 October 2016
  - Annual Review
  - Update the temperature heating range to 90-95°C where stated in the SOP
  - Removed the reference to AFCEE throughout SOP
  - Added current section 3.1 – reference to QAM for general definitions
  - Restructured and renumbered section 6.0
  - Added initial paragraph to section 6.0 regarding the documentation of equipment IDs
  - Revised the current sections 6.1 and 6.2 to reflect consistent verbiage and formatting as other SOPs
  - Added current section 7.6 Ultra Pure Peroxide reference
  - Added current footnote 1 to the section 8 table regarding soil preservation
  - Re-numbered previous footnote 1 to be footnote 2 to the section 8 table

- Updated section 9.1 and subsections to reflect current practices and verbiage
- Re-numbered Notes in section 9.3.3 to be Note 1 and Note 2
- Added LCSD required when an MS/MSD is not available to sections 9.3.2 and 9.3.3 Note 2
- Renumbered and updated section 11.1 and 11.2 to reflect current practices and verbiage
- Added current section 11.2
- Updated section 13.2 to reflect current practices and verbiage
- Added Strontium to Table 3
- Removed Titanium and Zirconium from Table 3

- Revision 8 dated 31 October 2015
  - Annual Review
  - Edited Sections 9.5.1 and 9.5.2 to clarify glass bead requirement
  - Added definition of reagent water
  - Updated Section 11.6.4 and 11.7.6 to reflect current practice
  - Removed Method exception 1 regarding method blank limits as it no longer applies
  - Added detail to training requirements for new analysts Section 13.3
  - Added note to Section 9.5.3 regarding precision requirements
  - Added note to Section 11.3 regarding recording of glass bead weights

- Revision 7 dated 31 March 2015
  - Annual Review
  - In Section 11.7.8 the section referenced was updated to 11.8
  - Updated spike standard name to MS spike 2 in Section 11.11.3
  - Formatting and grammar corrections throughout
  - Section 6.4 removed reference to calibrating digestion tubes
  - Section 6.6 changed name of filter paper to match current practice
  - Section 6.14 added to define computer systems used
  - Sections 7.7.3.1 and 7.7.3.2 combined
  - New Sections 7.7.3.2 - 7.7.3.4 added to define spikes used
  - Table of spike names and concentrations added to Section 7.7.3.4
  - Changed LIMS to TALS throughout
  - Section 8.2 changed storage temperature to ≤6 °C
  - Deleted Section 9.3, duplicated in 13.2
  - Added new Section 9.3 to address federal requirements
  - Rewrote Section 9.5
  - Changed Sections 9.6 – 9.8 to be subsections of the new 9.5
  - Rewrote Section 11.2.1
  - Removed method modification 2 because it referred to the analytical SOP
  - Created new method modification 2 explaining the 1-1.5 g sample aliquot
  - Section 11.3.2 changed required sample aliquot to 1-1.5 g to help avoid targeting
  - Rewrote Section 11.4 to define and explain the Incremental Sampling Method
  - Added new Section 11.5.3 to explain sample mixing
  - Section 11.7.5 added language to note regarding samples that require more than 10 mL of H2O2
  - Added detail into Sections 11.9.1 – 11.9.5
  - Folded Section 11.10.1 into 11.10

- Rewrote list of data to be entered into TALS in Section 11.10
- Rewrote Section 13.2 to match boilerplate
- Deleted flowcharts Figures 1 and 2
- Corrected element list in Table 2

- Revision 6 dated 31 March 2014
  - Annual Review
  - Formatting changes throughout document
  - Added to Section 11.7.5 to add additional peroxide to QC if added to samples
  - Updated section number in text to 11.8 in section 11.7.8
  - Added references for DoD QSM
  - Removed Attachment 2

- Revision 5 dated 04 March 2013
  - Section 7.7.3.1 Added DoD to the glass beads requirement
  - Section 11.11.2 Added that 5ml of water is added to the samples
  - Section 11.11.3 Changed spike name to 200.8 Cal-2
  - Updated spike level to 1.0ml in Table 3
  - Updated work instructions to current revision.
  - Formatting changes throughout document

- Revision 4 dated 3 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
  - Section 2.0 Added reference to Incremental Sampling Method
  - Section 6.4 Added 50 mL digestion tubes
  - Added introductory statement to section 7.0 regarding reagent purity
  - Section 7.1 Updated acceptable criteria for the reagent water
  - Section 9.7.2 Added LCS Incremental Sampling Method spike amounts
  - Section 9.8.2 Added MS/MSD Incremental Sampling Method spike amounts
  - Section 11.4 Updated sample amount for Incremental Sampling Method to 1 10g aliquot
  - Section 11.9 Added Incremental Sampling Method final volume

- Revision 3.5, dated 24 August 2011
  - A note has been added to section 9.8.3 for the addition of the LCS/MS spike before reagents.

Earlier revision histories have been archived and are available upon request.

Table 1.

Method 3050B Approved Analyte List for ICP/ICP-MS

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

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Table 2.

Soil LCS and MS/MSD Spikes for ICP

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (mg/L)
Aluminum	1,000	1,000	10.0
Antimony	100	100	1.0
Arsenic	200	200	2.0
Barium	200	200	2.0
Beryllium	100	100	1.0
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	100	100	1.0
Calcium	5,000	5,000	50
Chromium	100	100	1.0
Cobalt	100	100	1.0
Copper	100	100	1.0
Iron	1,000	1,000	10.0
Lead	100	100	1.0
Lithium	100	100	1.0
Magnesium	5,000	5,000	50
Manganese	100	100	1.0
Molybdenum	100	100	1.0
Nickel	100	100	1.0
Phosphorous	2,000	2,000	20
Potassium	5,000	5,000	50
Selenium	200	200	2.0
Silicon	200	200	2.0
Silver	5	5	0.05
Sodium	5,000	5,000	50
Strontium	100	100	1
Sulfur	1,000	1,000	10
Thallium	200	200	2
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	100	100	1.0
Zinc	50	50	0.50
Zirconium	50	50	0.50

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Table 3.

Soil LCS and MS/MSD Spikes for ICP-MS

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (µg/L)
Aluminum	400	400	4,000
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Iron	400	400	4,000
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Strontium	40	40	400
Thallium	20	20	200
Thorium	20	20	200
Tin	20	20	200
Tungsten	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Attachment 1

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with 5% HNO_3 according to the procedure described in SOP DV-IP-0005.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead or cobalt.
- New glassware can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves contain over 500 ppb of zinc.

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Title: Mercury in Solids by Cold Vapor Atomic Absorption

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS# 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A or 7471B.

1.2 Method 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, and sludge-type materials. All matrices require sample preparation prior to analysis. This is not an appropriate procedure for the digestion of tissues or other organic matrices, which require the use of EPA 245.6 instead.

1.3 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

1.4 The routine reporting limit for mercury in solid matrices is 17 µg/kg.

2.0 Summary of Method

A representative portion of the sample is digested in aqua regia in the first digestion cycle and potassium permanganate in the second cycle. Mercury is reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption of light at 253.7 nm is calibrated as a function of mercury concentration.

3.0 Definitions

3.1 Total Mercury: Inorganic forms of mercury are effectively dissolved by the acids used in the digestion. The potassium permanganate reagent breaks down organo-mercury compounds to inorganic forms that are detected by this procedure.

3.2 Aqua Regia: A 3:1 mixture of hydrochloric and nitric acids. This mixture is effective at dissolving metals in the solid form.

3.3 General Analytical Terms: Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Potassium permanganate “suitable for mercury determination” is specified because of the potential for mercury contamination in the reagent. In addition, potassium permanganate crystals will absorb mercury vapors from the air. Reagent bottles must be kept tightly closed to avoid contamination.

4.2 Potassium permanganate, in addition to breaking down organic compounds, also

eliminates possible interferences from sulfide. Concentrations as high as 20 ppm of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 ppm had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

NOTE: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be lessened by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe,

nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. A disposable face shield should be used when making up aqua regia.

5.3.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury Nitrate Solutions	Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydroxylamine Hydrochloride	Corrosive Poison	No OSHA PEL listed for this compound	Direct contact with skin or eyes causes irritation. May cause skin sensitization, an allergic reaction. Inhalation or ingestion may cause methemoglobinemia and resulting cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood), and labored breathing.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90 – 95 °C.

6.1.2 Mercury Autoanalyzers:

Teledyne M7500 and M7600 Mercury Analyzer with Autosampler

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable digestion tubes with caps from Environmental Express, volume accuracy verified to ASTM Class A standards at all gradations.

6.3.2 Disposable glass autosampler tubes, 16 mm x 100 mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated mechanical pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ± 1 °C at 95 °C (see SOP DV-QA-0001 for calibration details).

6.3.7 Glass beads, < 1 mm diameter, acid washed.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Primary and secondary standards used for data production are recorded in the Reagent Module of the Eurofins TestAmerica LIMS (TALS).

7.1 **Reagent water:** Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** Concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** Concentrated, trace metal grade or better.

7.4 **Aqua Regia:** Add 75 mL concentrated HCL and 25 mL concentrated HNO3 to a 100 mL container. Aqua Regia will be prepared immediately before use.

7.5 **Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO_3:**

7.5.1 Add 0.5 L of concentrated HNO_3 to a 50 L carboy partially filled with reagent water.

7.5.2 Dilute to 50 L with reagent water.

7.5.3 Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.6 **Stannous chloride solution ($SnCl_2$), Hg grade, 10% (w/v) per manufacturer's instructions:**

7.6.1 Place approximately 100 mL of reagent water into a 2 L volumetric flask.

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of $SnCl_2$ to the flask.

7.6.4 Mix the contents of the flask until the reagent is completely dissolved.

7.6.5 Bring solution to a final volume of 2 L with reagent water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (Hg grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate, 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Purchased Mercury Stock Solutions

7.9.1 Second source initial calibration verification (ICV) stock solution 100 mg/L (Hg ICV Stock).

7.9.2 Primary Mercury Calibration Standard Solution, 1,000 mg/L (Hg Ultra Prim).

7.10 Monthly Calibration Working Standard Solution, 10 mg/L (Hg Mnth Spike)

7.10.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.10.2 Pipet 1.0 mL of the 1,000 mg/L primary mercury calibration standard solution (Section 7.9.2) into the flask.

7.10.3 Dilute to the mark on the flask with 1% HNO_3.

7.10.4 Stopper the flask and shake to mix.

7.10.5 Transfer the solution to a 125 mL Nalgene bottle.

7.10.6 Document the preparation of the solution in the Reagent Module in TALS.

7.10.7 Prepare this solution fresh monthly or more often if necessary.

7.11 Daily Calibration Working Spike, 100 µg/L (Hg Daily Spk)

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL volumetric flask.

7.11.2 Add 1.0 mL of the 10 mg/L Calibration Working Standard (see Section 7.10).

7.11.3 Bring the solution to a final volume of 100.0 mL.

7.11.4 Stopper and mix thoroughly.

7.11.5 Document the preparation in the Reagent Module in TALS.

7.11.6 Prepare this solution each day prior to calibration.

7.12 Initial Calibration (ICAL) Standards

The initial calibration standards are prepared directly in the digestion tubes as follow:

ICAL	Daily Calibration Working Spike (mL)	1% HNO_3 (mL)	Final Conc. (µg/L)
Blank	0.0	5.0	0.0
Std 1	0.10	4.9	0.20
Std 2	0.25	4.75	0.50
Std 3	0.50	4.5	1.0
Std 4	1.0	4.0	2.0
Std 5	2.5	2.5	5.0
Std 6	5.0	0.0	10.

7.13 Second-Source Initial Calibration Verification Intermediate Standard, 400 µg/L (Hg Biwk ICV)

Add 40 µL of the 1000 mg/L ICV Standard to a 100 mL volumetric flask partially filled with 1% HNO_3 and dilute to the mark. Record this information in the Reagent Module in TALS.

7.14 Second-Source Initial Calibration Verification (ICV) Daily Working Standard, 4.00 µg/L (Hg Soil ICV)

Add 0.5 mL of the 400 µg/L ICV Intermediate Standard (see Section 7.13) to a soil digestion tube and add 4.5 mL of 1% HNO_3. Record this information in the Reagent Module in TALS.

7.15 Continuing Calibration Verification (CCV) Standards, 5 µg/L (Hg Soil CCV)

7.15.1 The CCVs are prepared exactly as the 5.0 µg/L ICAL standard shown above (see Section 7.12).

7.15.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.16 Laboratory Control Sample (LCS), 333 µg/kg

7.16.1 The LCS is prepared in an empty digestion tube or 0.6 g of glass beads (< 1 mm) are used if required.

7.16.2 Add 2.0 mL of the 100 µg/L Daily Calibration Working Spike (see Section 7.11) to a digestion tube. See Section 9.4 for additional detail.

7.16.3 This is equivalent to 4.0 µg/L, which is the concentration that appears on the raw data printout from the instruments.

7.17 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 333 µg/kg

MS/MSD pairs are spiked in the same manner as the LCS (see section 7.16) and prepared in the same manner as the samples, using 0.6 g of sample.

7.18 Reporting Limit (CRA) Check Standard, 0.2 µg/L (Hg Soil RL)

7.18.1 Add 0.1 mL of 100 µg/L Daily Calibration Working Spike (see section 7.11) and 4.9 mL of reagent water to a digestion tube.

7.18.2 This is equivalent to a 0.2 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	10 grams	Cool, ≤ 6 ºC	28 Days	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Eurofins TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for the DoD/DOE QSM (5.0 – 5.3) unless otherwise stated. Any deviation or exceptions

from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3 Method Blank (MB)

The MB consists of an empty vessel (or with glass beads for DoD/DOE projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ RL or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

NOTE: DOD QSM 5 does not allow this exception. Results may not be reported without a valid

method blank unless sample cannot be re-prepared or re-analyzed.

9.4 Laboratory Control Sample (LCS), 333 µg/kg

The preparation of the LCS is described in Section 7.16. At least one LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. DoD requires when there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Maximum control limits for LCS recoveries are 80 - 120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see Policy DV-QA-003P for further details on establishing control limits).

NOTE: DOD QSM 5 Solid matrix LCS Limits are 80 - 124%.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

NOTE: Reporting sample results with failing LCS recoveries is not acceptable for DOD QSM 5 unless samples cannot be re-prepared or reanalyzed.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 333 µg/kg

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recovery must fall within 75-125%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

NOTE: DOD QSM 5 Solid matrix MS/MSD Limits are 80-124%.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the linear range and the MS/MSD results are above the linear range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within linear range and the MS/MSD results

are above the linear range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE 1: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE 2: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

9.6 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 2 for details).

NOTE: DOD QSM 5: Performed only when required by the project and the SD or PDS fails. Must be documented with an NCM and included in the case narrative.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure. Prepare digestion tubes containing volumes of standards required for each tube as listed in Section 7.

10.3.2 Weigh 0.5 – 0.6 g of each well-homogenized sample into a sample digestion tube. See SOP DV-QA-0023 for additional information on subsampling.

10.3.3 If preparing Incremental Samples a 3 g sample aliquot is required. This aliquot will be prepared by utilizing the procedure described in *Incremental Sampling Methodology for Soils and Sediments* (DV-OP-0013). Divide the 3 g aliquot into five 0.6 g samples. Digest the five individual aliquots and combine them back into one after adding the sodium chloride-hydroxylamine hydrochloride reagent. All batch QC samples must also be processed in this fashion.

10.3.4 Prepare a MB, LCS, MS, and MSD for each batch. The MB is either an empty digestion tube or is prepared by placing 0.6 g of glass beads in a digestion tube, depending on client requirements. The LCS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube. The MS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube containing a second aliquot of the chosen matrix sample. The MSD is prepared in the same manner as the MS using a third aliquot of the chosen sample.

NOTE: The spike must be added after the sample aliquot but before the addition of reagents.

10.3.5 Add 5.0 mL of reagent water to all un-spiked field samples and the method blanks. Add 3.0 mL of reagent water to the LCS, MS and MSD.

10.3.6 Add 5.0 mL of Aqua Regia to each tube.

10.3.7 Heat for 2 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS.

10.3.8 Allow the samples and standards to cool at room temperature.

10.3.9 Add 19 mL of reagent water.

10.3.10 Add 15 mL of 5% potassium permanganate solution. A purple color must persist for at least 15 minutes. If the color does not persist, the sample must be re-prepared using a smaller sample aliquot.

NOTE: It is important that equal volumes of the potassium

permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors.

10.3.11 Cap the samples and standards and heat for 30 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS. The analyst will verify that a purple color persists or a black precipitate is present after the thirty minutes of heating. If this is not true, the digestion must be repeated using a smaller sample aliquot.

10.3.12 Allow the samples and standards to cool at room temperature.

10.3.13 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate. Verify that the volume is at 50 mL.

10.3.14 For samples aliquoted using the Incremental Sampling Method combine the 5 individual sample cups for each sample and QC into one marked 250 mL container.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis. Preparation of calibration standards is described in Section 7.12.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.3 Detailed information regarding calibration models and calculations can be found in Corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

10.4.4 Initial Calibration (ICAL)

10.4.4.1 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4.4.2 Calibrate using six standards and a blank (see Section 7.12).

10.4.4.3 The calibration curve must have a correlation coefficient (r^2) ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. The calibration curve is processed with an unweighted linear regression.

10.4.4.4 Record the microAbsorbance (µAbs.) for the 10 ppb standard

in the instrument maintenance log.

NOTE: It is not acceptable to reject calibration points for this method.

10.4.5 Initial and Continuing Calibration Blanks

10.4.5.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DOD QSM 5 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.5.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. As just noted, DOD QSM 5 requires that results for blanks must be less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.6 Initial Calibration Verification (ICV), 4.0 µg/L (Hg Soil ICV)

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV recovery must be within 90 - 110%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

10.4.7 Reporting Limit Check Standard (CRA), 0.2 µg/L (Hg Soil RL)

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for QSM 5.1).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, the instrument recalibrated, and associated samples reanalyzed.

10.4.8 Continuing Calibration Verification (CCV), 5.0 µg/L (Hg Soil CCV)

Calibration accuracy is monitored during the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: The CCV recovery must be within 80 - 120% except for QSM 5.0 where the CCV recovery limits are 90 - 110%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be reanalyzed once without modification to the instrument‘s operating conditions. If the reanalyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.4.9 CCV Acceptance Criteria for sample run under a DOD QSM 5.0 program

CCVs must have a percent recovery of 90 - 110%. If the CCV fails the following options are available: Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately (within an hour) analyzed 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.

10.5 Sample Analysis

10.5.1 Set up the instrument and autosampler according to the manufacturer’s

instructions.

10.5.2 Allow the samples to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.3 Pipet 10 mL of each sample and calibration standard into a disposable test tube for analysis

10.5.4 Analyze the standards and samples according to the manufacturer's instructions.

10.5.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples with mercury concentrations that exceed the highest calibration standard.

NOTE: The instrument auto-dilutes samples. Any samples that require greater than a 10x dilution MUST be diluted manually.

10.5.6 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be reanalyzed.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB.

10.5.8 The analytical sequence listed below must be followed. Refer to *Quality Control* Section 9.0 for quality control limits.

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.0 Calculations / Data Reduction

Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{spike concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration = mg/kg or L

$$= \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mL)

Note: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4 Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

11.4.1 Sample data entered into the preparation batch in TALS, which includes the batch number, list of samples, preparation analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes).

11.4.2 Raw data (direct instrument printout as a PDF) with the analyst name and all required calibration information.

11.5 Reporting

11.5.1 Standard units for reporting solid sample results are mg/kg.

11.5.2 If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor,

and the data may require flagging.

11.5.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.5.4 All associated data are entered or uploaded into TALS as required.

11.5.5 Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.5.6 The initial data review is performed by the analyst while the second-level data review is performed by the area supervisor or designee. Both reviews are documented in TALS. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. MDL verifications are performed quarterly.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require such verification. A blank matrix is spiked at 1 - 2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for

each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee) the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and Policy DV-HS-001P, *Waste Management Plan*.

14.1 The following waste streams are produced when this method is carried out:

14.1.1 Aqueous Acidic (Metals) - Corrosive - (J)

14.1.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, February 2007, Method 7471B (Mercury).

15.3 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013.

15.4 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.2, 2018.

15.5 U.S.EPA Statement of Work for Inorganic Analysis, ILMO3.0

16.0 Method Modifications:

Item	Method	Modification
1	7471A	An additional QC analysis, RL verification, is added
2	7471A	Methods 7470A and 7471A state that working standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory had developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing (PT) results for samples that were prepared and analyzed within 24 hours, but on successive days • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
3	7471A	Chapter 1 of SW-846 specifies the use of reagent water with a purity equivalent of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
4	7471A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of

Item	Method	Modification
		interest at or above ½ the reporting limit.
5	7471B	Method 7471B uses reagent water for the method blank. Eurofins TestAmerica Denver is currently using glass beads when required.
6	7471B	Section 11.1 requires 50 mL of reagent water to be added to the sample with 15 mL of Potassium permanganate. Eurofins TestAmerica Denver utilizes digestion tubes which do not allow for 50 mL of reagent water. 19 mL of reagent water is currently being added.
7	7471A	Section 7.1 specifies triplicate 0.2 g portions of sample for solids analysis. Eurofins TestAmerica Denver instead utilizes a 0.5 - 0.6 g weight range in order to avoid targeting of specific weights and to comply with the requirements of the most recent method revision.

17.0 Attachments

Attachment 1: Summary of Quality Control Requirements
Attachment 2: MSA Guidance
Attachment 3: Instrument Maintenance
Attachment 4: Troubleshooting Guide

18.0 Revision History

- Revision 14 dated 02 October 2020
  - Updated standard concentration and spiking amount in Section 7.13.
  - Added instrument M7600 to Section 6.1.2

- Revision 13 dated 19 September 2019
  - Updated standard concentration and spiking amount in Section 7.13.

- Revision 12 dated 31 May 2019
  - Annual technical review.
  - Minor formatting and language corrections throughout
  - Removed Section 3.1 due to redundancy
  - Updated amount of reagent water added in Section 10.3.5 to 3 mL.
  - Added QSM 5.1 CRA criteria to Section 10.4.7 and Attachment #1.
  - Removed Section 11.4.3 regarding data checklists
  - Removed reference to QSM 4.2 and added reference to QSM 5.2 in Section 15

- Revision 11 dated 31 May 2018
  - Annual Technical review.
  - Updated the spike amount used in Section 10.3.4 to 2ml.

- Revision 10 dated 31 May 2017
  - Updated the spike concentration for the LCS, MS and MSD in sections 7.16 and 7.17

  - Updated true value for LCS and MS/MSD in sections 9.4 and 9.5
  - Added DoD requirement of LCSD if no MS/MSD to sections 9.4 and 9.5
  - Added clarification to section 9.5 regarding parent and MS/MSD samples that are “over range”

- Revision 9 dated 01 Feb 2017
  - Annual Technical Review
  - Added a paragraph to Section 6.0 regarding the documentation of equipment IDs
  - Updated Section 7.4 to make the aqua regia in 100 mL container
  - Added Note 2 to Section 9.5 Referencing MS/MSD Policy Memoranda
  - Added a paragraph to Section 10.3 regarding the documentation of equipment IDs
  - Updated language in Section 12.1 regarding MDL studies
  - Added current Section 12.2 defining LOQVs

- Revision 8 dated 28 Feb 2016
  - Annual Technical Review
  - Added current Section 3.1
  - Removed reference to AFCEE program in Section 9.3
  - Modified/Updated Section 9.5 – Corrective Actions for MS/MSD failures
  - Updated Sections 12.2 & 12.3 to be more consistent with other SOPs
  - Archived revision histories 2010 and earlier – available upon request

- Revision 7 dated 28 Feb 2015
  - Annual Technical Review
  - Removed reference to purging sample headspace from Section 4.4, outdated.
  - Section 4.6 – Removed reference to method 245.6
  - Changed MSDS to SDS
  - Removed references to “Cetac Only”
  - Added Section 7.5.3 regarding recording of data in TALS
  - Added TALS reagent IDs to standard names
  - Corrected concentrations of Cal Stds
  - Corrected Section references
  - Changed units of RL std to water units
  - Changed minimum sample volume to 10 g to accord with corporate policy
  - Added DoD V5 references
  - Changed sample aliquot to 0.5 – 0.6 g
  - Deleted references to adjust volume with 1% HNO3
  - Updated temperature of digestion to 95 ± 3 oC
  - Deleted Section 10.4.4.3 (Redundant)
  - Changed “counts” in Section 10.4.4.4 to microAbsorbance
  - Removed “Approximately” from ICV true values
  - Removed GC references from Section 10.4.9
  - Removed references to “resloping”
  - Added initial CCV/CCB pair to sequence

- Revision 6 dated 28 Feb 2014
  - Annual Technical Review
  - Removed references to Serial Dilutions and Post Digestion Spikes
  - Section 10.4 for incremental sampling was merged into Section 10.3
  - Updated Section 10.4.4.1 and 10.4.4.2 to note DOD QSM 5 criteria

- Updated Section 10.4.7 to note DOD QSM 5 CCV criteria is 90-110.
- Updated Attachment 2 for ICB,CCB and CCV criteria to DOD QSM 5
- Added Attachment 5 for Troubleshooting

- Revision 5 dated 15 July 2013
  - Annual Technical Review
  - Correction to formatting
  - Changed reference to Standards Log to Reagent Module in the LIMS
  - Added General Analytical Terms information to definition section
  - Edited section 7.6, 10.4.8, 12.1 & 12.2 to reflect current practices.
  - Changed RL reference in sections 7.18, 10.5.6, 10.6.8 and Attachment 2 to CRA
  - Removed bullet point 5 under 11.5
  - Removed Attachment 3 and renumbered the subsequent attachments
  - Corrected references date for section 15.2
  - Added Texas TRRP to section 12.1

- Revision 4 dated 30 September 2012
  - Clarified the language in Section 9.4 to be one LCS per batch.
  - Modified Section 7.16.2 to refer to Section 10.4.4 for additional detail.

- Revision 3.2 dated 13 July 2012
  - Updated Sections 7.6 and 7.7 to state Hg grade reagents are used
  - Updated Sections 10.4.11 to include a note about bringing samples to a final volume before the sample is mixed
  - Updated Section 10.5.4 and Attachment 2 to control calibrations blanks to ½ the RL.
  - Added Sections 10.5.3.5 to record the number of counts for the 10 ppb standard
  - Formatting and grammatical changes throughout

- Revision 3.1 dated 03 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling throughout document
  - Annual Technical Review
  - Section 1.3 Added Incremental Sampling Method statement to SOP
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated section 9.5 and Attachment 2 for method blank control criteria
  - Section 10.2 Added Incremental Sampling Method preparation amount
  - Section 10.2.12 Added Incremental Sampling Method combination procedure
  - Section 10.2.13 Added Incremental Sampling Method final volume
  - Added dilution note to Section 10.3.5
  - Updated section 12.0 to reflect current laboratory practices

Earlier revision histories have been archived and are available upon request.

Attachment 1

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Immediately following ICAL	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICV	Following ICB	90 - 110% recovery	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
RL Check Standard (CRA)	Following the ICV	50 - 150% recovery 80 - 120% for QSM 5.1	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery. 90 - 110% for QSM 5.0	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ≤ ½ RL Sample results greater than 10% the blank concentration are acceptable.	Redigest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 80 - 120%	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75 - 125%	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75 - 125% recovery or in-house control limits; RPD ≤ 20%	See Corrective Action for Matrix Spike.

Attachment 2

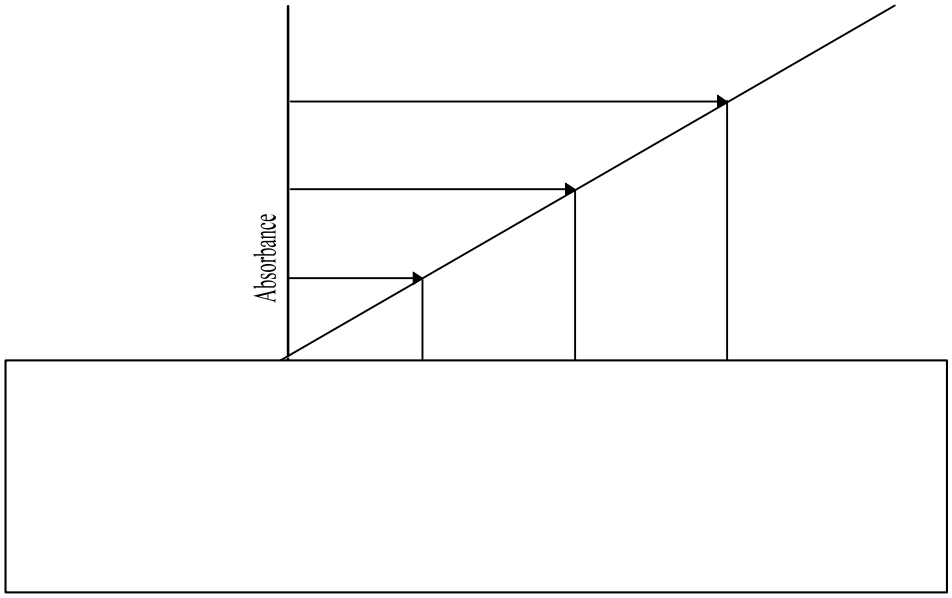
MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 3

Instrument Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time and instrument number, then identify the problem and corrective action in the maintenance log. When the instrument is returned to service, record the return to service, the date, and any tests performed to verify proper operation.

The following preventative maintenance procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

Attachment 4

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell

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Title: Mercury in Water by Cold Vapor Atomic Absorption (CVAA) [SW 7470A]

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A.

1.2 Method 7470 is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts.

1.3 All matrices require sample preparation prior to analysis.

1.4 The final reporting limit is 0.0002 mg/L (0.2 µg/L), except for TCLP leachates that have a 0.002 mg/L (2 µg/L) reporting limit.

2.0 Summary of Method

This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration). All sample data are uploaded to the Eurofins TestAmerica LIMS (TALS).

3.0 Definitions

3.1 Dissolved Metals: Those elements that pass through a 0.45-µm membrane. (Sample is acidified after filtration).

3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

3.3 Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Assurance Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper also has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides will require dilution. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents,

and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.3 Potassium permanganate is a strong oxidizing agent. It is incompatible with and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.

Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90-95°C.

6.1.2 Mercury Auto-analyzers: The laboratory currently uses a QuickTrace™ M7500 and M7600 with Autosamplers.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable 50 mL digestion tubes with caps. Accuracy at 30 mL verified to ±3% gravimetrically prior to use (by lot). See DV-QA-0008 for more information regarding volume verifications.

6.3.2 Disposable glass test tubes, 16 mm x 100 mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ±1 °C at 95 °C (see SOP DV-QA-0001 for calibration details).

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the

determination. Suggested reagent and standard recipes are listed below. Alternate weights and volumes may be used as long as the final concentrations are maintained as listed and the details are recorded in the Reagent module in TALS. All standard concentrations listed below refer to the on-instrument concentration except where otherwise noted.

7.1 **Reagent water:** Must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** concentrated, trace metal grade or better.

7.4 **Sulfuric acid (H_2SO_4):** concentrated, trace metal grade or better.

7.5 **Reagent Blank:** This blank solution is used as the Calibration Blank (STD0), Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and as the starting solution for the Method Blanks (MBs). It is made as follows:

Add 0.5 L of concentrated HNO_3 to a 50-liter carboy partially filled with reagent water. Dilute to 50 L with reagent water. Mix carefully. Record the acid lot numbers and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.6 **Stannous Chloride Solution, Hg grade, 10% (w/v) per manufacturer's (CETAC) instructions**

7.6.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.6.2 Add 200 g of $SnCl_2$ to the flask.

7.6.3 Add deionized water until the total weight is 2000 g.

7.6.4 Place the jar in a fume hood and slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.5 Close the jar and agitate until the reagent is dissolved (prepare every 2 weeks).

7.7 **Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):**

7.7.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.7.2 Add 240 g of NaCl and 240 g of hydroxylamine hydrochloride (Hg grade) to the jar.

7.7.3 Add deionized water until the total weight is 2480 g.

7.7.4 Close the jar and agitate until the reagent is dissolved. (prepare

annually).

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate ($KMnO_4$), 5% solution (w/v):

7.8.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.8.2 Add 100 g of $KMnO_4$ (Hg grade) to the jar.

7.8.3 Add deionized water until the total weight is 2100 g.

7.8.4 Close the jar and agitate until the reagent is dissolved (prepare annually).

7.9 Potassium persulfate ($K_2S_2O_8$), 5% solution (w/v):

7.9.1 Place approximately 1000 g of deionized water in a tared 2-L HDPE jar on a top-loading balance.

7.9.2 Add 100 g of $K_2S_2O_8$ (Hg grade) to the jar.

7.9.3 Add deionized water until the total weight is 2100 g.

7.9.4 Close the jar and agitate until the reagent is dissolved (prepare annually).

7.10 Purchased Mercury Stock Solutions

7.10.1 Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.10.2 Second-source Mercury Standard (Hg ICV Stock), 1000 mg/L. This standard is obtained from a different vendor than the Primary Mercury Calibration Standard.

7.11 Calibration Working Standard Solution (Hg Month Spike), 10 mg/L.

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.11.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution (see Section 7.10.1) into the flask.

7.11.3 Dilute to the mark on the flask with Reagent Blank.

7.11.4 Stopper the flask and shake to mix.

7.11.5 Transfer the solution to a 125 mL Nalgene bottle.

7.11.6 Document the preparation of the solution in the Reagent Module in TALS.

7.11.7 Prepare this solution fresh monthly or more often if necessary.

7.12 Daily Calibration Working Solution (Hg Daily Spk), 100 µg/L

7.12.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.12.2 Pipet 1.00 mL of the 10 mg/L Calibration Working Standard solution (see Section 7.11) into the flask.

7.12.3 Dilute to the mark on the flask with the Reagent Blank solution (final volume of 100.0 mL).

7.12.4 Stopper the flask and shake to mix.

7.12.5 Transfer the solution to a 125 mL Nalgene bottle.

7.12.6 Document the preparation of the solution in the Reagent Module in TALS.

7.13 Daily Initial Calibration (ICAL) Standards.

7.13.1 To each of seven 50 mL digestion tubes, add approximately 30 mL of the Reagent Blank solution.

7.13.2 For each calibration level, add the appropriate amount of Daily Calibration Working Solution to the tube as indicated in the following table. The final concentration for each calibration level is listed in the following table:

Daily ICAL Standards

Calibration Level	Volume of Daily Calibration Working Solution (100 µg/L) Added (mL)	Final Hg Concentration (µg/L)
1 (Hg STD1 0.1)	0.03	0.06
2 (Hg STD2 0.2)	0.06	0.12
3 (Hg STD3 0.5)	0.15	0.3
4 (Hg STD4 1.0)	0.3	0.6
5 (Hg STD5 2.0)	0.6	1.2
6 (Hg STD6 5.0)	1.5	3.0
7 (Hg STD7 10.0)	3.0	6.0

7.13.3 Close each tube and swirl to mix.

7.13.4 Prepare the calibration standards as samples.

7.13.5 Document the preparation of the solutions in the Reagent Module in TALS.

7.13.6 Prepare the calibration solutions each day prior to calibration.

7.13.7 The calibration blank is titled STD0 in TALS.

7.14 Continuing Calibration Verification Standard (Hg H20 CCV), 3.0 µg/L.

7.14.1 The CCV is prepared exactly as the 3.0 µg/L calibration standard, and from the same source. Refer to Section 7.13.

7.14.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.15 Second-Source Initial Calibration Verification Intermediate Standard (Hg Biwk ICV), 400 µg/L.

Add 0.04 mL of the 1000 mg/L ICV stock standard (see Section 7.10.2) to a 100 mL volumetric flask partially filled with the Reagent Blank solution and dilute to the mark. Record this information in the Reagent Module in TALS. This standard is made biweekly.

7.16 Second-Source Initial Calibration Verification Daily Working Standard (Hg H20 ICV), 2.4 µg /L.

Add 0.3 mL of the 400 µg/L ICV intermediate standard (see Section 7.15) to a 50 mL digestion tube filled with 30 mL of Reagent Blank. Prepare as a sample. Record this information in the Reagent Module in TALS.

7.17 Laboratory Control Sample (LCS), 3 µg/L

The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Standard (Section 7.12) to 30 mL of Reagent Blank in a digestion tube. The LCS goes through the same digestion process as the samples.

7.18 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 3 µg/L

7.18.1 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution (Section 7.12) to a digestion tube containing a second 30-mL aliquot of the selected sample.

7.18.2 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

7.18.3 The MS and MSD go through the same digestion process as the samples.

7.19 Reporting Limit (CRA) Check Standard (Hg H20 RL), 0.12 µg/L

The 0.12 µg/L calibration standard is analyzed as a sample to verify the reporting limit. Denoted as CRA in the run sequence.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	HDPE or glass	50 mL	HNO_3, pH < 2	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. See Table 1 for a summary of these requirements. This procedure meets all criteria for DoD QSM 5.0 or 5.1 unless otherwise stated. Any deviations or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC

9.2.1 Preparation Batch

A group of no more than 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.2.2 Method Blank (MB)

The method blank consists of Reagent Blank containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

For DoD V5 the method blank is controlled to < ½ LOQ or 10% of the amount measured in any sample or 10% of the regulatory limit, whichever is greater.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was <u>not</u> detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.2.3 Laboratory Control Sample (LCS)

The LCS is a blank to which a known concentration of the target analyte has been added. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Maximum control limits for LCS recoveries for Method 7470A are 80-120%. In-house control

limits based on three standard deviations of the mean of past results are used as long as they are at least as tight as the limits in the methods (see TestAmerica Denver Policy DV-QA-003P for further details on establishing control limits).

For DoD V5.0 or V5.1 the QSM Appendix C limits are required.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.2.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a second aliquot of a selected field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported for project-related samples only. Spiking levels are provided in Attachment 1. When the MS/MSD concentration is above the linear range; the MS/MSD and parent sample **MUST** be re-analyzed at a dilution. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5.0 or 5.1 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the

established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.2.5 Serial Dilution

Some programs require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the initial sample concentration is at least 25x the MDL concentration (or 50x the LOQ for DoD).

Corrective Action: If the control limit is not met, all associated sample results must be qualified and the failure addressed in the narrative.

9.2.6 Post-Digestion Spike

Some programs require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.3 mL of the 100 µg/L Daily Calibration Working Solution to 10 mL of sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion

spike are 80 to 120%.

Corrective Action: If the acceptance criteria are not met, all associated sample results must be qualified.

9.2.7 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 3)

9.3 Instrument QC

9.3.1 Initial Calibration (ICAL)

9.3.1.1 Detailed information regarding calibration models and calculations can be found in Corporate Policy CA-Q-S-005, *Calibration Curves & Selection of Calibration Points*.

9.3.1.2 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. All calibration standards and calibration QC samples will be recorded in prep batches and prepared as samples. The instrument calibration date and time must be included in the raw data.

9.3.1.3 Calibrate using seven standards and a blank. The concentration levels are listed in Attachment 1.

NOTE: It is generally not acceptable to reject calibration points for this method.

9.3.1.4 The calibration curve must have a correlation coefficient of ≥ 0.995 for an unweighted linear regression or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

9.3.1.5 Record the microabsorbance for the 10 ppb standard in the instrument maintenance logbook.

9.3.2 Initial and Continuing Calibration Blank (ICB/CCB)

9.3.2.1 An initial calibration blank is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DoD QSM 5.0 or 5.1 requires control of blanks to a

concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.2.2 Continuing calibration blanks are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. Some programs require that blanks be less than 2x the MDL or less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.3.3 Initial Calibration Verification (ICV), 2.4 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV result must be within 10% of the true value.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

9.3.4 Reporting Limit Check Standard (CRA), 0.12 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value. For DoDV5.1 the recovery is within 20%

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, and the instrument

recalibrated.

9.3.5 Continuing Calibration Verification (CCV), 3.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run. The CCV must be a mid-range standard at a concentration other than that of the ICV.

Acceptance Criteria: The CCV result must fall within 20% of the true value.

For DoD V5 the CCV result must be within 10%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be re-analyzed once without modification to the instrument's operating conditions. If the re-analyzed CCV is found to be in control, the CCV analysis must be repeated a second time with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

9.3.6 Linear Range

TAL Denver does not report values greater than the highest standard (10 µg/L) used for calibration. Any sample concentration greater than 90% of the highest standard will be diluted. The calibration curve is validated by running 3 check standards, 0.12 µg/L (CRA) , 3 ug/L (CCV), and 2.4 µg/L (ICV), during the analytical run. No further linear range study is warranted.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

NOTE: Record the IDs of all pipettes and thermometers in the TALS batch record.

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure.

10.3.2 Transfer 30.0 mL of well mixed sample to a clean sample digestion tube. The calibration standards may be prepared in duplicate to ensure sufficient volume to complete the analytical sequence. Additional aliquots of CCV and CCB solution may have to be prepared for larger sample runs to ensure that CCV and CCB samples bracket every 10 samples in the analytical sequence.

10.3.3 Prepare an MB, LCS, MS, and MSD for each batch.

10.3.3.1 The MB consists of 30.0 mL of Reagent Blank.

10.3.3.2 The LCS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to 30 mL of Reagent Blank in a digestion tube.

10.3.3.3 The MS is prepared by adding 1.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second 30-mL aliquot of the selected sample.

10.3.3.4 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

10.3.4 Add 1.5 mL of concentrated H_2SO_4 and 0.75 mL of concentrated HNO_3 to the samples in the digestion tubes, mixing after each addition.

10.3.5 Add 4.5 mL of 5% potassium permanganate solution to each sample. For samples high in organic materials or chlorides, dilute the sample until the purple color persists for at least 15 minutes.

10.3.6 Add 2.4 mL of potassium persulfate solution, cap the vial, and heat for two hours at 90 - 95°C. Record the start and stop times and the initial and final temperatures on the bench sheet. Verify that a purple color persists or a black precipitate is present after the two hours of heating. If this is not true, repeat the digestion using a smaller aliquot of sample.

NOTE: Record both the observed and corrected temperatures on the bench sheet.

10.3.7 Allow the samples and standards to cool at room temperature.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration by starting the gas, lamp, heater, and sample pump (approximately 30 minutes of warm-up is required).

10.4.3 The mercury analyzer method uses external standard calibration. Use of an internal standard for this method is not appropriate.

10.5 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument-operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.5.1 When ready to begin analysis, add 1.8 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).

10.5.2 Add additional Reagent Blank to the samples, QC samples and calibration standards to bring the final volume of each sample to 50 mL.

10.5.3 Aliquot each sample and calibration standard into a disposable test tube for analysis.

10.5.4 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples that are within 10% of the highest calibration standard.

10.5.5 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.5.6 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB.

10.5.8 The following analytical sequence must be used for Method 7470A. Refer to Quality Control Section 9.0 and Attachment 2 for quality control

criteria to apply to Method 7470A.

Instrument Calibration
ICV
ICB
CRA
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete the run.
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- **Recovery of the analyte in the matrix spike is <50%;**
- **The concentration of the analyte does not exceed the regulatory level; and**
- **The concentration of the analyte is within 20% of the regulatory level.**
- **The reporting and matrix spike levels for TCLP analyses are detailed in Attachment 1. Attachment 3 provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.**

10.5.10 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

10.5.11 See Attachment 5 for guidelines for minimizing contamination of samples and standards. See Attachments 4 and 6 for guidance on troubleshooting and preventive maintenance.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves & Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

11.2 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$

11.3 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.4 Concentration = Hg concentration (µg/L) = $\frac{C \times V_1 \times D}{V_2}$

Where:

C = Concentration (µg/L) from instrument readout

D = Instrument dilution factor

V_1 = Final volume in liters after sample preparation

V_2 = Initial volume of sample digested in liters

11.5 Appropriate factors must be applied to sample values if dilutions are performed.

11.6 Sample results should be reported with up to three significant figures in accordance with the Eurofins TestAmerica significant figure policy (DV-QA-004P).

11.7 Documentation and Record Management

11.7.1 All sample data is uploaded to TALS. All sample preparation and analytical batch information, including the batch number(s), list of samples, preparation analyst and date, instrument analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes) is recorded in TALS.

11.7.2 Raw data is scanned or saved directly as a PDF and is attached to the analytical batch in TALS.

11.7.3 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

11.7.4 If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor and the data may require flagging.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Aqueous Acidic (Metals) - Corrosive - Waste Stream J

14.2.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

15.3 U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

15.4 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 5.0, 7/2013

16.0 Method Modifications:

Item	Method	Modification
1	EPA 7470A	Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
2	EPA 7470A	This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."
3	EPA 7470A	Methods 7470A and 7471A state that working mercury standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory has developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • **Successful proficiency testing PT results for samples that were prepared and analyzed within 24 hours, but on successive days (e.g., ERA WP-66);** • **Successful analysis of true NIST mercury standards within every analytical batch; and** • **A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."**
4	EPA 7470A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above ½ the reporting limit.

17.0 Attachments

Attachment 1: Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels
Attachment 2: Summary of Quality Control Requirements
Attachment 3: MSA Guidance
Attachment 4: Troubleshooting Guide
Attachment 5: Contamination Control Guidelines
Attachment 6: Preventative Maintenance
Table 1: DOD QSM 5.0/5.1 QC Criteria for CVAA/Mercury

18.0 Revision History

- Revision 10 dated October 02, 2020
  - Annual review
  - Added M7600 instrument to Section 6.1.2

- Revision 9 dated September 19, 2019
  - Annual review

- Revision 8 dated August 31, 2018
  - Annual review
  - Removed vendor from Section 7.10.1
  - Updated standard concentration in Section 7.10.2
  - Updated standard concentration and spiking amount in Section 7.15.

- Revision 7 dated August 31, 2018
  - Annual review
  - Removed reference to use of an auto diluter in sections 6.1.2 and 10.5.4.
  - Added control criteria for DoDV5.1 in Section 9.3.4 for the CRA.
  - Updated Attachment 2 for DoDV5.1 CRA and DoDV5 CCV control limits.

- Revision 6 dated August 31, 2017
  - Annual review
  - Added reference to QSM 5.1 throughout the SOP
  - Added support equipment ID documentation requirement to section 6.0
  - Clarified batch definition to “no more than 20 samples” to section 9.2.1
  - Added reference to LCSD requirement to sections 9.2.3 and 9.2.4
  - Added current section 12.2 LOQV information

- Revision 5 dated August 31, 2016
  - Annual review
  - Removed all references to AFCEE
  - Added comment to section 7.6 to prepare Stannous Chloride Solution every two weeks
  - Added comment to sections 7.7 Sodium chloride-hydroxylamine hydrochloride solution, 7.8 Potassium permanganate, and 7.9 Potassium persulfate to prepare annually
  - Updated section 9.2.4 MS/MSD acceptance criteria and corrective actions
  - Added note to 10.3 to document IDs of pipettes and thermometers
  - Added note to 10.3.6 to record observed and corrected temperatures
  - Updated a three subsection of section 12 to reflect current practices
  - Update Attachment 2 for ICB/CCB for DoD to <LOD
  - Removed revision histories for 2010 and earlier (available upon request)

- Revision 4 dated July 31, 2015
  - Annual review
  - Added 0.1 standard to Section 7.13.2
  - Updated MB control limit to ½ LOQ for DoD
  - Removed 20% DoD control for CRI from Section 9.3.4
  - Added DoD V5 spiking requirement for MDLV to Section 12.1
  - Updated Section 12.2 DOC to use LCS’s

- Added 0.1 standard to Attachment 1
- Added TALS reference to Section 2.0
- Added instructions to Section 7.5 regarding making reagent blank
- Added TALS reagent IDs
- Change LIMS to TALS throughout
- Added Linear range section 9.3.6
- Corrected Section 11.7 to match current practice
- Removed figures one and two
- Revised reagent Sections 7.6-7.9 to match current practice
- Changed Sections 7.13-7.19 to reflect new procedure
- Updated procedures in Section 10 to better meet traceability requirements

- Revision 3 dated July 31, 2014
  - Annual review
  - Added DoD V5 requirements to Sections 9.2.2, 9.2,3, 9.2.4, 9.3.2.1 and 9.3.5.
  - Added table 1 - DOD QSM 5.0 QC Criteria for CVAA/Mercury

- Revision 2 dated July 15, 2013
  - Annual review
  - Changes section 7.6 and 12 to reflect current practices
  - Remove reference to Standards Log to Reagent Module in the LIMS in section 7.11.6, 7.12.6, 7.13.4, 7.15 & 7.16.
  - Changed “RL” to “CRA” in sections 7.19, 9.3.4, 10.5.8
  - Added CRA (RL Standard) to Attachment 2
  - Removed Attachment 3 and re-number subsequent attachments
  - Clarified first bullet point under 10.5.9
  - Corrected references date for section 15.2
  - Added Texas TRRP to section 12.1

- Revision 1.2 dated July 13, 2012
  - Updated Sections 7.6 and 7.7 to state Hg reagents are used
  - Updated Sections 9.3.2.1 and 9.3.2.2 to control calibration blanks to ½ RL
  - Added Section 9.3.1.5 to record the counts for the 10 ppb high standard
  - Updated Sections 10.5.2 to bring samples to a final volume of 45 mL with 1% HNO_3
  - Formatting and grammatical changes

- Revision 1.1 dated February 03, 2012
  - Annual technical review
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated Section 9.1.2 and Attachment 2 for Method Blank acceptance criteria
  - Added dilution note to Section 10.3.4
  - Updated section 12.0 to reflect current laboratory practice
  - Removed Leeman instrument and replaced Nitrogen with Argon for Attachment 7

- Revision 1.0 dated 23 August 2011
  - Updated Section 7.15 ICV Intermediate Standard to 400ug/l
  - Updated Section 7.16 ICV Working Standard level to 4ug/l
  - Updated Section 9.2.3 ICV true value to 4ug/l
  - Updated Section 10.3.8 ICV and ICB run order

- Revision 0.5 dated 25 April 2011
  - Removed all references to the FIMS Analyzer
  - Sections 6.1 and 6.3 were updated to reflect the use of digestion blocks from water baths.
  - The reagent amounts were updated to reflect using a 30ml aliquot from 10ml.
  - Section 10.3.2 was updated to show a final volume of 40ml.

- Revision 0.4 dated 07 February 2011
  - Revised section 10 to reflect use of calibrated digestion tubes and calibration standard volumes
  - Revised supplies list
  - Revised section 6.2 to include reference to Master List of Documents, Software and Hardware
  - Added section 11.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”

Earlier revision histories have been archived and are available upon request.

Attachment 1

Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels (µg/L)

	Value at Instrument	Final Value
Standard Aqueous RL	0.12	0.2
Std 0	0	0
Std 1	0.06	0.1
Std 2	0.12	0.2
Std 3	0.3	0.5
Std 4	0.6	1.0
Std 5	1.5	2.0
Std 6	3.0	5.0
Std 7	6.0	10.0
ICV	2.4	4.0
CCV	3.0	5.0
LCS	3.0	5.0
Aqueous MS	3.0	5.0

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Attachment 2

Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run.	90 - 110% recovery	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.3).
RL Check Standard (CRA)	Following the ICV	50-150% recovery, 80-120% for DoDV5.1	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICB	Beginning of every analytical run, immediately following the ICAL.	Absolute value must be < ½ RL, <LOD for DoD	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.3.2).
CCV	Every 10 samples and at the end of the run.	80 - 120% recovery, 90-110 for DoDV5	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.3.5).
CCB	Immediately following each CCV.	Absolute value must be < ½ RL, <LOD for DoD	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.3.2).
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ½ RL Sample results greater than 10x the blank concentration are acceptable.	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 80-120% recovery.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.2.3).
Matrix Spike	One per 10 samples preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 75-125% recovery	In the absence of client-specific requirements, flag the data (see Section 9.2.4).
Matrix Spike Duplicate	See Matrix Spike	In-house 3 standard deviation control limits, not to exceed 20% RPD	See Corrective Action for Matrix Spike.

Attachment 3

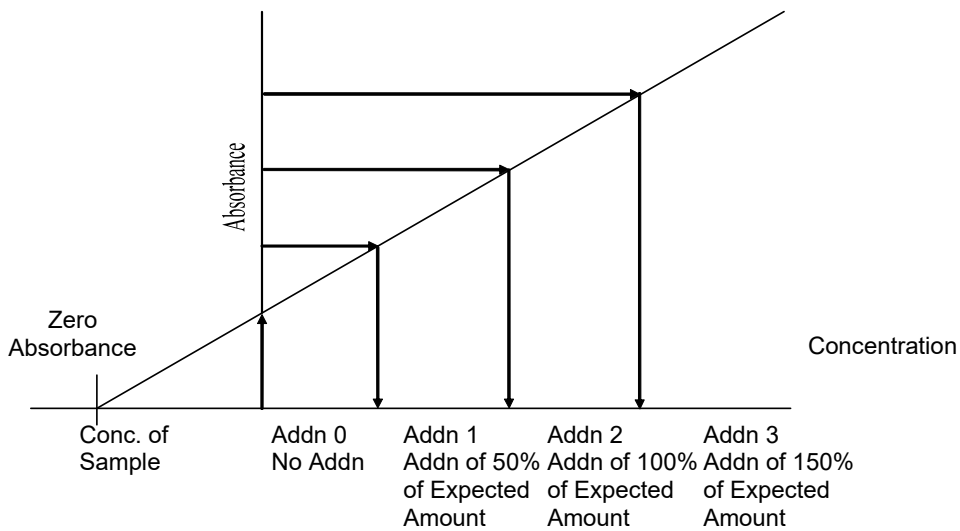
MSA Guidance

Method of Standard Addition (MSA)

Four equal volume aliquots of sample are measured and known amounts of standards are added to three of the aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration, and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of an analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. The absorbance (or response) is plotted on the vertical axis versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. The correlation coefficient (r) and the x-intercept (where y=0) of the curve are calculated. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 4

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

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Attachment 5

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered gloves should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- If an unusually high sample is analyzed, segregate the glassware and soak with sulfuric acid prior to routine cleaning.

Attachment 6

Preventative Maintenance

A maintenance logbook is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time, and instrument number; describe the problem; and explain the corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational:

Cold Vapor Atomic Absorption (CETAC Analyzers)

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

Table 1
DOD QSM 5.0 & 5.1 QC Criteria for CVAA/Mercury

QSM 5.0 5.1 Table 7. Inorganic Analysis by CVAA/Mercury	
Requirement	**DoD QSM 5.0, QSM 5.1 and DOE QSAS 3.0**
Initial Calibration (ICAL)	Measure a minimum of 5 standards and a calibration blank daily and $r^2 \geq 0.99$ No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Run second-source standard once after each ICAL and prior to sample analysis. Must be within ± 10% of expected value. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV after every 10 samples, and at the end of the analysis sequence: Reported analyte within ± 10% of expected value If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV. Or Immediately (within one hour) 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 analytes(s) in all samples since the last acceptable CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank	Analyze calibration blank before analyzing samples, after every 10 field samples, and at the end of the analysis sequence. No analytes detected > LOD. Correct any problems, then re-prep and reanalyze the calibration blank and analyze all samples since the last acceptable calibration blank. Failures due to carryover may not require an ICAL.
Method Blank	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.

Table 1
DOD QSM 5.0 and 5.1 QC Criteria for CVAA/Mercury
(Continued)

QSM 5.0 and 5.1Table 7. Inorganic Analysis by CVAA/Mercury	
DoD QSM 5.0, QSM 5.1 and DOE QSAS 3.0	
LCS	One per prep batch. Recovery must meet DoD QSM limits. If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.
Matrix Spike (MS)	One MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. If acceptance criteria are not met, apply J-flag. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.

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Title: Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on EPA Method 6020. The reporting limits for all elements are listed in Attachment 1.

1.2 Method 6020 lists fifteen elements approved for analysis by ICP-MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). This procedure has been developed for twenty elements and additional elements may be included provided that the method performance criteria presented in Sections 9 and 12 are met. However, project approval may be required from the controlling agencies for compliance testing beyond the elements included in the promulgated methods.

1.3 The procedure is applicable to the analysis of acid digested waters, sediments, sludges and soils. Standard reporting limits are listed in Attachment 1 for water and soil. The preliminary acid digestion for aqueous samples is described in SOP DV-IP-0014, and the digestion procedure for solids is given in SOP DV-IP-0015.

2.0 Summary of Method

2.1 Aqueous digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into a radiofrequency (RF) plasma. There the sample is decomposed and desolvated.

2.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer capable of providing a mass resolution better than or equal to 0.9 amu (see Section 3) peak width at 10% of the peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

2.3 A collision/reaction cell utilizing He and (optionally) H_2 gases is used to remove molecular interferences. As the ion beam passes through the cell chamber, a diffuse cloud of He or H_2 gas is injected into its path. Collisions between the ions and the atoms in the gas deflect and remove interferences. See Section 4.2.3 for more information on interferent removal.

2.4 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference corrections must include compensation for background ions contributed by the plasma gas, reagents, and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Attachment 2. Interference equations may vary or be unnecessary depending on the instrument setup and choice of collision/reaction gas.

2.5 Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices. Internal standard assignments are listed in Attachment 4.

3.0 Definitions

3.1 Atomic Mass Unit (amu) – Obsolete term replaced by "unified atomic mass unit (u)" or "dalton (Da)", which denotes a small unit of mass that is used to express atomic and molecular masses. It is defined to be 1/12 of the mass of one atom of carbon-12.

3.2 Batch – The batch is a set of up to 20 samples of the same or similar matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. See Policy DV-QA-003P for further details.

3.3 Dissolved Metals - Those elements which pass through a 0.45-µm membrane filter (sample is acidified after filtration).

3.4 Total Metals - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5 Total Recoverable Metals - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6 Instrument Detection Limit (IDL) - See Section 12.3.

3.7 Sensitivity - The slope of the analytical curve (i.e., the functional relationship between raw instrument signal and the concentration).

3.8 Tuning Solution - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9 Initial Calibration Verification / Quality Control Standard (ICV) - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at concentrations other than those used for instrument calibration.

3.10 Continuing Calibration Verification (CCV) - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11 Interference Check Standard (ICS) - A solution containing both interfering and analyte elements of known concentration that is used to verify background and interelement correction factors.

3.12 Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB) - A multi-element standard of known concentrations that is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13 **Reagent Blank -** High purity water containing the same acid matrix as the calibration standards that is carried through the entire digestion process.

3.14 **Calibration Blank -** High purity water acidified with the same acid concentrations present in the standards. Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15 **Method Detection Limit (MDL) -** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

3.16 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Elemental Isobaric Interferences

Elemental isobaric interferences in the ICP-MS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software and by the careful selection of isotopes for analysis.

4.2 Isobaric Molecular Interferences

4.2.1 Polyatomic interferences are derived from the plasma gas, reagents or sample matrix. Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Attachment 3 lists isobaric interferences which might possibly affect required analytes. These molecular interferences are minimized by use of the collision cell utilizing He and/or H_2 gases. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.2.2 Chloride in samples can produce low recoveries for antimony and silver. If chloride interference is a concern, 1% HCl can be added during digestion, but calibration standards must be adjusted to include 1% HCl also. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

4.2.3 Collision cell interference removal works both by causing the interfering molecular ion to dissociate and by reducing the kinetic energy of the ion. The latter is termed Kinetic Energy Discrimination (KED), and is the primary mechanism for interference removal. Polyatomic ions are larger than elemental ions and so collide with the helium atoms in the collision cell more frequently than the smaller elemental ions. Each collision

reduces the energy of the ion, so the molecular ions lose energy more quickly. At the end of the collision cell a positive voltage plate prevents passage of the now low energy molecular ions. Thus, the interference is eliminated because the molecular ions do not reach the detector.

4.3 Doubly Charged Ion Interferences

Doubly charged elemental ion interferences are possible in cases where the second ionization potential of the element is significantly below the first ionization potential for argon (15.7 eV). If a doubly charged ion is formed, it will cause a response at half of its elemental mass, potentially causing interference. Most elements have high enough second ionization potentials that formation of doubly charged ions is not an issue. The percentage of doubly charged ions being formed in the plasma is monitored on a daily basis during the instrument tuning process.

4.4 Physical Interferences

4.4.1 Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.4.2 Internal standards should be added at a level to give approximately 100,000 – 20,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 50 amu of the mass of the measured analyte.

4.4.3 Matrix effects are monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing Method 6020, the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. If they fall outside this window, a five-fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample is reanalyzed.

4.4.4 Memory effects or carry-over can occur when there are large relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

5.0 <u>Safety</u>

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP-MS plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) capable of providing resolution less than or equal to 0.9 amu at 10% peak height and 1.0 amu at 5% peak height in the mass range from 6 - 253 with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. The ICP-MS must be equipped with a collision cell for the removal of molecular interferences.

6.1.2 A four-channel peristaltic pump.

6.1.3 Autosampler with autosampler tubes.

6.1.4 Appropriate water cooling device.

6.2 Supplies

6.2.1 Argon, Helium and Hydrogen gas: High purity grade (99.99%).

6.2.2 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.3 Class A volumetric flasks and graduated cylinders

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards must be entered into the TestAmerica LIMS (TALS) Reagent Module. Reagents that are not used for calculating results may either be recorded in the Reagent Module or may be entered into batch worksheets.

7.1 Storage and Shelf-Life

7.1.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standards stored at concentrations as received from the vendor and mid-level dilutions must be replaced prior to the expiration date assigned by the vendor. If no expiration date is provided, the stocks and mid-level standards may be

stored for up to one year. They must be replaced sooner if verification from an independent source indicates a problem.

7.1.2 Working standards, i.e., all standards at concentrations ready to analyze on the ICP-MS (except tuning mixes, ICSA and ICSAB mixes, which are received at ready-to-use concentrations) are prepared fresh daily.

7.1.3 For more information on standard storage and shelf-life, see SOP DV-QA-0015.

7.2 Standards

Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in TALS.

7.2.1 Tuning Solution

The parent tuning solution is purchased as a custom multi-element mix. The elements and concentrations of the constituents are shown in Attachment 7. Prepare the working Tuning Solution at least every 6 months as detailed below.

7.2.1.1 Obtain a clean 1 L volumetric flask

7.2.1.2 Place 500 mL of reagent blank in the flask

7.2.1.3 Pipette 1 mL of the Tuning Solution Stock into the flask

7.2.1.4 For the Agilent 7700 instruments add 50 µL of a 500 mg/L Mg solution and 30 µL of a 1,000 mg/L Be solution to the flask.

7.2.1.5 Dilute to volume with the appropriate reagent blank (see Section 7.3). Stopper and mix.

7.2.2 P/A factor solution

7.2.2.1 The Pulse/Analog (P/A) solution is used to monitor the correlation between the Pulse counting and Analog modes of the electron multiplier. The diluted solution must be prepared at least every 6 months at different concentrations depending on the current instrument conditions. Multiple dilutions may be required to cover the required intensity range for all elements.

7.2.2.2 The P/A solution may be commercially purchased as a custom multi-element mix. See Attachment 7 for a list of the constituents and concentrations.

7.2.2.3 Prepare and use the P/A solution as recommended by the instrument manufacturer. The P/A solution should be analyzed daily.

7.2.3 Calibration Standard

Stock calibration standards are purchased as custom multi-element mixes or as single element solutions. Each day of analysis, the standards are diluted to working levels using reagent blank (see Section 7.3). The concentrations are given in Attachment 11. Prepare the Daily Working Calibration Standard as shown.

7.2.3.1 Daily Working Calibration Standard for Instruments 077 and 078 (ms 77 cal std)

7.2.3.1.1 Obtain a clean 250 mL volumetric flask.

7.2.3.1.2 Place 100 mL of reagent blank in the flask.

7.2.3.1.3 Pipette 0.25 ml of each MS CALSTD 1 thru 6

7.2.3.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.4 Initial Calibration Verification (ICV) Standard

The ICV stock is from a source different than the source for the calibration standards. Each day of analysis, the ICV standards are prepared in the reagent blank to the concentrations shown in Attachment 11. Prepare the ICV as shown below:

7.2.4.1 Initial Calibration Verification Standard for Instruments 077 and 078 (MS 77 ICV)

7.2.4.1.1 Obtain a clean 250 mL volumetric flask.

7.2.4.1.2 Place 100 mL of reagent blank in the flask.

7.2.4.1.3 Pipette 0.1 mL of each MS ICV Mix 1 thru 6

7.2.4.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.5 Continuing Calibration Verification (CCV) Standard

The CCV is prepared from the same source as the calibration standards. The CCV standards are prepared fresh each day of analysis in the reagent blank. The concentration is shown in Attachment 11.

7.2.5.1 Continuing Calibration Verification for Instruments 077 and 078 (ms 77 ccv)

7.2.5.2 Using a graduated cylinder pour 100 ml each of the MS CAL Daily and reagent blank for a final volume of 200ml.

7.2.6 Reporting Limit Standards (RL/CRI)

The reporting limit standards are prepared fresh daily from the same stock as the calibration standards using the reagent blank. The analyte concentrations must be less than or equal to the respective reporting limits. Multiple solutions may be required in order to satisfy all of the project and client specific reporting limits. Alternate reporting limit concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents module in TALS. Prepare the Reporting Limit Standard for the Agilent 7700 as detailed below.

7.2.6.1 RL Standard for Instruments 077 and 078 (ms 77 RL)

7.2.6.1.1 Obtain a clean 50 mL volumetric flask.

7.2.6.1.2 Place 30 mL of reagent blank in the flask.

7.2.6.1.3 Pipette 0.5 mL of the ms 77 cal std solution into the flask.

7.2.6.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.7 Daily Linear Range Standard

The Linear Range standard is prepared daily from the same stock as the calibration standards using reagent blank.

7.2.7.1 Daily Linear Range Standard for Instruments 077 and 078 (MS 77 LR STD)

7.2.7.1.1 Obtain a clean 500 mL volumetric flask.

7.2.7.1.2 Place 50 mL of reagent blank in the flask.

7.2.7.1.3 Pipette 1.0 mL of a 1,000 mg/L As standard into the flask.

7.2.7.1.4 Pipette 2.5 mL of a 1,000 mg/L Ba standard into the flask.

7.2.7.1.5 Pipette 1.0 mL of a 1,000 mg/L Be standard into the flask.

7.2.7.1.6 Pipette 1.0 mL of a 1,000 mg/L Cd standard into the flask.

7.2.7.1.7 Pipette 1.0 mL of a 1,000 mg/L Co standard into the flask.

7.2.7.1.8 Pipette 2.5 mL of a 1,000 mg/L Cr standard into the flask.

7.2.7.1.9 Pipette 2.5 mL of a 1,000 mg/L Cu standard into the flask.

7.2.7.1.10 Pipette 5.0 mL of a 1,000 mg/L Mn standard into the flask.

7.2.7.1.11 Pipette 1.0 mL of a 1,000 mg/L Mo standard into the flask.

7.2.7.1.12 Pipette 2.5 mL of a 1,000 mg/L Ni standard into the flask.

7.2.7.1.13 Pipette 2.5 mL of a 1,000 mg/L Pb standard into the flask.

7.2.7.1.14 Pipette 0.5 mL of a 1,000 mg/L Sb standard into the flask.

7.2.7.1.15 Pipette 1.0 mL of a 1,000 mg/L Se standard into the flask.

7.2.7.1.16 Pipette 1.0 mL of a 1,000 mg/L Sn standard into the flask.

7.2.7.1.17 Pipette 0.5 mL of a 1,000 mg/L Tl standard into the flask.

7.2.7.1.18 Pipette 1.0 mL of a 1,000 mg/L U standard into the flask.

7.2.7.1.19 Pipette 1.0 mL of a 1,000 mg/L V standard into the flask.

7.2.7.1.20 Pipette 2.5 mL of a 1,000 mg/L Zn standard into the flask.

7.2.7.1.21 Pipette 0.05 mL of a 10,000 mg/L Th standard into the flask.

7.2.7.1.22 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.8 Internal Standard (IS) Solution (77 I.S. / MS I.S. INT)

The internal standard solution is added continuously by peristaltic pump through a mixing tee. The concentrations and components are specified in Attachment 4. Prepare the IS solution at least every 6 months as follows:

7.2.8.1 Obtain a clean 250 mL volumetric flask.

7.2.8.2 Place 100 mL of reagent blank in the flask.

7.2.8.3 Pipette 1.2 mL of the 1,000 mg/L Ge Standard into the flask.

7.2.8.4 Pipette 0.4 mL of the 1,000 mg/L Ho Standard into the flask.

7.2.8.5 Pipette 0.4 mL of the 1,000 mg/L In Standard into the flask.

7.2.8.6 Pipette 0.75 mL of the 1,000 mg/L Sc Standard into the flask.

7.2.8.7 Pipette 1.5 mL of the 1,000 mg/L ^{6}Li Standard into the flask.

7.2.8.8 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9 Interference Check Standard Solutions (ICSA / ICSAB)

The interference check standard solution (ICSA) and the spiked interference check standard solution (ICSAB) are prepared weekly as follows:

7.2.9.1 ICSA Standard (ms 77 icsa / MS ICSA)

7.2.9.1.1 Obtain a clean 100 mL volumetric flask.

7.2.9.1.2 Place 50 mL of reagent blank in the flask.

7.2.9.1.3 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9.2 ICSAB Standard (MS 77 ICSAB / MS ICSAB)

7.2.9.2.1 Obtain a clean 100 mL volumetric flask.

7.2.9.2.2 Place 50 mL of reagent blank in the flask.

7.2.9.2.3 Pipette 0.1 mL of each MS ICV Mix 1 thru 6 stock standards into the flask.

7.2.9.2.4 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.2.5 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.3 Reagents

7.3.1 Reagent Water - Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

7.3.2 Reagent Blank - Agilent 7700, 2% HNO_3/0.05% HCL Carefully dilute 40 mL of concentrated HNO_3 and 10 mL of concentrated HCL in 2.0 L of reagent water. This solution is used to dilute samples and it is used for calibration blanks.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Water[3]	HDPE	50 mL	HNO_3, pH < 2	180 Days	SW-846
Soil	Glass	4 oz	Cool, ≤ 6 °C[4]	180 Days	SW-846

[1] SW-846 specifies a holding time but does not specify a preservation for solid samples for total metals. TAL-Denver refrigerates these samples.

[2] Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Holding Times are calculated from the date the sample was collected.

[3] Water samples collected for dissolved elements are filtered immediately on-site by the sampler before adding preservative.

[4] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply. Quality control requirements are summarized in Attachment 10.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 and 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

For aqueous and soil samples, the method blank consists of reagent water that has been processed in the same manner as the samples. For soil samples analyzed under the DoD QSM, the method blank consists of <1 mm glass beads that have been processed in the same manner as the samples. One method blank must be processed with each preparation batch.

Acceptance Criteria: Method blank results are acceptable if the concentration for each analyte of interest is less than ½ the reporting limit (RL). For DoD QSM 5.0/5.1 the control limit is less than ½ LOQ. In the absence of project specific reporting limits, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable.

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination should be investigated to determine if the problem can be minimized or eliminated. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- The same analyte was not detected in the associated samples;
- The method blank concentration is less than 1/10 of the measured concentration of any sample in the batch;
- The method blank concentration is less than 1/10 the specified regulatory limit; or
- The analyte is a common laboratory contaminant (e.g., iron, zinc) less than the RL. Note that some programs do not recognize common lab contaminants.

If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative and the client must be notified.

9.3 Laboratory Control Sample (LCS)

The LCS consists of reagent water that is spiked with the analytes of interest at the project specific action level or, when lacking specific action levels, at approximately the mid-point of the calibration range (summarized in Attachment 11). For soil samples analyzed under the DoD QSM, the LCS consists of < 1 mm glass beads that have been spiked with the analytes of interest and processed in the same manner as the samples. One LCS must be processed for each preparation batch.

Acceptance Criteria: LCS control limits are based on three standard deviations of past laboratory results or program specific requirements. limits must not exceed 80-120%. The control limits are maintained in TALS. For DoD QSM 5.0 the laboratory must use QSM Appendix C Limits for batch control if project limits are not specified.

Corrective Action: If the LCS recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reanalyzed. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. If project requirements allow this exception, the data may be accepted with qualifiers, an

NCM must be generated, and the failure narrated in the final report.

9.4 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

The MS is prepared by taking a second aliquot of a selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 11). An MSD is prepared by taking a third aliquot of the selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 11). The MS and MSD are processed in the same manner as the samples. One MS/MSD pair must be processed for each preparation batch. Some programs require that matrix spikes can be performed only on project samples, and that the samples to be used are identified on the chain of custody form. The spike concentration should be the same level as the LCS.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.

- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.

- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE 1: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE 2: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE 3: According to the guidelines established in Section 8.4 of Method 1311, *Toxicity Characteristic Leachate Procedure (TCLP)*, if the MS/MSD recoveries from a TCLP batch are below 50%, do not exceed the regulatory limit, but are within 20% of the regulatory limit, the Method of Standard Additions (MSA) must be used to quantitate the sample results. If an MSA study is performed, the results must be calculated by hand using the MSA spreadsheet found at R:\QA\Edit\FORMS\Metals. See Attachment 9 for the TCLP regulatory limits. MSA instructions can be found in SOP DV-MT-0019, Attachment 7.

9.5 Interference Check Solutions (ICSA/ICSAB)

The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Attachment 5. The interference check solutions must be analyzed at the beginning of every analytical run and once every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences.

Acceptance Criteria: The results for the spiked elements (AB portion) must be ±20% of the expected value. In addition, the internal standard recoveries for both the ICSA and AB must be within 30-120%. Some programs have control limits for the non-spiked elements in the ICSA. Please check the client specific requirements. For DoD QSM 5.0 the ICSA for non-spiked analytes is controlled to less than the absolute value of the LOD unless they are a verified impurity.

Corrective Action: If the ICSAB results exceed the 20% limit or the internal standard is out in the ICSA or AB, then the analysis sequence must be terminated. For DoD QSM 5.0 if the ICSA is outside of the control limits for the non-spiked elements the sequence must also be terminated. The problem must be investigated and fixed. The ICSA and all affected samples must be re-analyzed.

NOTE: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (e.g., ICPAES), acceptance criteria will be applied at that level and the data accepted.

9.6 Internal Standards Evaluation for Samples

The IS recovery in samples must be between 30% and 120% of the intensity of the calibration blank. If sample IS recoveries fall outside of these criteria, a five-fold (1:4) dilution must be performed, the dilution analyzed, and the same acceptance criteria applied.

9.7 Serial Dilution

One serial five-fold dilution should be analyzed per preparation batch. If the analyte concentration is within the linear range of the instrument and sufficiently high (generally, a factor of 10 above the MDL in the diluted sample), the serial dilution must agree to within 10% of the original analysis. If not, an interference effect is suspected and a flag is applied to the data. The failure must be addressed in the final report narrative. Samples identified as blanks should not be used for the serial dilution. For DoD QSM 5.0 if the parent sample result is greater than 50X the LOQ prior to dilution then the serial dilution must agree within 10%. If the acceptance criteria are not met then the parent sample is flagged “J”.

9.8 Post-Digestion Spike Addition (PDS)

A PDS is performed for each batch. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 - 125% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected. Typically the concentration of the PDS is 200 µg/L for each element except silver which is spiked at 50 µg/L. For DoD QSM 5.0 if the parent sample concentration is less than 50x the LOQ prior to dilution then the PDS must recover within 80-120%. If the recovery is outside of the control limits for a given element then the parent sample is flagged “J”.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

10.3 Instrument Maintenance

See Section 20 in the QAM.

10.4 Instrument Troubleshooting

See Attachment 12

10.5 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0014 and DV-IP-0015).

10.6 Calibration

10.6.1 Instrument Start Up

Set up the instrument according to manufacturer's operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning. It is recommended that the instrument be flushed with the ICSA solution to help condition the cones and improve stability. Allow the instrument time to rinse completely before tuning the instrument.

10.6.2 Oxide\Doubly Charge Performance Check

With the sample probe in the Tune solution verify that the oxides and doubly charged ions are less the 3% by running the Tune report.

10.6.3 Instrument Tuning / Mass Calibration

Tune the instrument with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 5 times. Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day. The Tune must be rerun if any changes are made to the instrument during the day. If either of the following conditions fails the instrument setup must be re-evaluated and the solution rerun.

Mass Calibration Check: The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

Mass Resolution Check: The resolution at 5% peak height should be approximately 0.75amu.

10.6.4 Initial Calibration

The ICP-MS is calibrated each day of operation using a blank and a single standard (see Section 7.2.2.1). At least three integrations are employed. The validity of the calibration is determined by the

subsequent calibration verifications, which are performed at concentrations as described in the next sections.

10.6.5 Second-Source Initial Calibration Verification (ICV)

A 40 µg/L ICV standard (see Section 7.2.4) is analyzed immediately after the initial calibration. This is a standard obtained from a different vendor than the standard used for calibration.

Acceptance Criteria: The ICV recovery must be within 90-110% and the internal standard recoveries must be within 30-120% of the calibration blank. The ICV can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICV results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.6 Calibration Blank

An initial calibration blank (ICB) is analyzed after the ICV. Continuing calibration blanks (CCBs) are analyzed after each continuing calibration verification. The appropriate reagent blank is used for the blanks.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. In addition, the internal standard recoveries must be within 30-120% of the associated calibration blank. Client specific requirements take precedence. For example, DoD QSM 5.0 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the calibration blank exceeds acceptance limits, then the possibility of instrument contamination should be examined, particularly the possibility of carry-over from high level samples. The blank can be reanalyzed, and if successful, analysis can continue. However, samples tested after high-level samples should be retested. If the reanalysis is not successful, then the analysis should be terminated. After the problem is corrected, recalibrate and reanalyze all samples tested since the last acceptable CCB.

10.6.7 Reporting Limit (RL/CRI) Verification Standard

Because the ICP-MS calibration does not include multiple calibration levels, an independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. The RL verification standard (see Section 7.2.6) is analyzed after the daily ICB.

Acceptance Criteria: For standard projects, the results should be within 50% of the expected value. Some programs may require tighter control. For DoD QSM 5.0 the control limits are 80-120%.

Corrective Action: If the RL verification fails to meet acceptance limits, data for the associated samples must be assessed. For example, if the results are high, consider blank contamination, and if the results are low, consider MDL verifications. At a minimum, sample results must be qualified in the final report. For DoD QSM 5.0, if the low-level standard does not meet the limits when spiked at the required project RL, the run sequence must be terminated.

10.6.8 Continuing Calibration Verification (CCV) Standard

A 50 µg/L CCV standard (see Section 7.2.5) is analyzed after every set of ten samples or every 2 hours, whichever is most frequent, and at the end of the analytical sequence.

Acceptance Criteria: The CCV recovery must be within 90-110%. In addition, the IS recovery must be within 30-120% of the calibration blank. If CCV results are not within these limits, the CCV can be reanalyzed, but it must be successful twice in succession or further corrective action must be taken.

Corrective Action: If the CCV fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCV.

10.7 Sample Analysis

10.7.1 Report the average of at least three integrations for all field and QC samples analyzed.

10.7.2 Flush the system with the rinse blank for a minimum of 30 seconds between samples and standards during the analytical run.

10.7.3 Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element. See Attachment 3 for examples.

10.7.4 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte. Dod QSM 5.0 requires that samples be diluted and reanalyzed if they are above the daily linear range check standard. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the RL. (The sample should be diluted to the approximate midrange of the analytical curve.)

10.7.5 The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization / Warm-Up
Tune instrument
Perform mass calibration
Perform resolution check
Validate tuning criteria
Calibration blank
Calibration standard
ICV
ICB
RL verification standard
ICSA
ICSAB
Daily Linear Range Verification Standard (if required – See Section 12.5)
CCV
CCB
10 Samples (which can include all sample types)
CCV
CCB
Reslope
CCV
CCB

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 ICV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}}\right) \times 100\%$$

11.3 CCV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}}\right) \times 100\%$$

11.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{SSR - SR}}{\text{SA}}\right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the detection limit, use SR = 0 for the purpose of calculating %R.

11.5 The relative percent difference (RPD) between sample duplicates is calculated according to the following equation:

$$RPD = \frac{|orig.\ sample\ value - dup.\ sample\ value|}{|(orig.\ sample\ value + dup.\ sample\ value)/2|} \times 100$$

11.6 The final concentration for an aqueous sample is calculated as follows:

$$\text{Result } (\mu\text{g/L}) = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration from instrument readout, ppb (ug/L)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

11.7 The concentration determined in digested solid samples when reported on a dry weight basis is as follows:

$$\text{Result } (\mu\text{g/kg}) = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration from instrument readout, ppb (ug/L)
D = Instrument dilution factor
V = Final volume in liters after sample preparation

W = Weight, in Kg, of wet sample digested

S = Percent solids/100

11.8 Sample data are reviewed by the analyst (Level 1 data review) and documented on the data review checklist. The data package is then submitted for level 2 review by another analyst or data reviewer. Second level review is documented on the same checklist initiated by the analyst. The data review process is explained in SOP DV-QA-0020.

11.9 **Trivalent Chromium:** See Work Instruction WI-DV-0092 *Calculation Methods* for additional details.

Total Chromium – Hexavalent Chromium

11.10 Reporting

11.10.1 Reporting units are ug/L for water samples and ug/Kg for solid samples.

11.10.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.10.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.10.4 All associated data are entered or uploaded into the LIMS as required.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.11 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard.

12.2.1 Prepare an MDLV standard at 2-4 times the calculated MDL concentration.

12.2.2 Analyze the MDLV standard immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

12.2.3 The calculated MDL is verified if the MDLV standard is detected, nominal signal to noise ratio > 3, under routine instrument conditions.

12.2.4 If the first MDLV is not detected, re-prepare the MDLV standard at twice the original concentration and analyze. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.4 Instrument Detection Limit Study

Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each analyte used for analysis in accordance with Policy DV-QA-014.

12.4.1 Pour out seven undigested calibration blanks and run them on three non-consecutive days.

12.4.2 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.4.3 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.5 Linear Dynamic Range (LDR)

12.5.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

12.5.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.5.3 The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards are required for the initial and on-going studies, and one of the levels must be at the upper end of the range. The highest concentration must be within 10% of the stated concentration.

12.5.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

12.5.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.6 Linear Range Verification (LRA/LRC)

The LDRs should be verified whenever, in the judgment of the analyst, a change in the analytical performance caused by either a change in instrument hardware or operating conditions would dictate the necessity to re-establish them. Some programs (e.g., USACE) require verification of linear ranges in each analytical run. As described in Section 7.2.7, a lower concentration is used for the daily check than is used for the quarterly determination.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the Linear Range Verification standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. Alternatively, results that do not exceed the level of the highest calibration standard may be accepted and reported.

12.7 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.7.1 Four aliquots of an LCS are analyzed using the same instrumental conditions and procedures used to analyze samples. The analyst must employ ICV's from four distinct analytical sequences. Using these four LCSs demonstrates the analyst's ability to optimize and calibrate the instrument and to prepare analytical solutions. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.7.2 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test

need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.7.3 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.8 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 **Aqueous Acidic (Metals) -** Corrosive - Waste Stream J

14.2.2 **Expired reagents and standards –** Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994.

15.1.2 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.3 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.4 Method 3050B, Acid Digestion of Sediments, Sludges and soils, Rev. 2, December 1996.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, March 2005.

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, October 2010.

15.4 U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

15.5 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6020	Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.
2	EPA 6020	The results of the calibration blank as well as all other blanks must be less than ½ the reporting limit, not 3 times the instrument IDL unless otherwise specified by a program or client.
3	EPA 6020	Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.
4	EPA 6020	The serial dilution results are evaluated when the original result is greater than 100 times the MDL rather than 100 times the concentration in the reagent blank.
5	EPA 6020	Corrective action for a PDS failure will be limited to flagging the PDS indicating the failed analyte and the recovery rather than diluting and reanalyzing the sample.
6	EPA 6020	Internal standard recoveries are based on the intensities of the internal standards in the most recent calibration blank rather than the intensities of the internal standards in the initial calibration standard.
7	EPA 6020	The internal standard recoveries for the interference check solutions and CCB's will be controlled to 30-120% of the calibration blank just as the samples and not 80-120%

Item	Method	Modification
8	EPA 6020	Method 6020 states that the dilution test is applicable if the matrix sample is at least 100x the reagent blank. TestAmerica uses the tighter limit of 50X the MDL.
9	EPA 6020	The Mass resolution criteria in method 200.8 is used to satisfy the criteria for method 6020

17.0 Attachments

Attachment 1: Standard Reporting Limits for Water and Soil
Attachment 2: Recommended Elemental Equations
Attachment 3: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes
Attachment 4: Internal Standards and Corresponding Metals
Attachment 5: Interference Check Sample Components and Concentrations
Attachment 6: Suggested Mass Choices
Attachment 7: Tuning Solution and P/A Solution
Attachment 8: Suggested Tuning and Response Factor Criteria
Attachment 9: TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Attachment 10: Summary of Quality Control Requirements
Attachment 11: Calibration, Calibration Verification, and Spike Concentrations
Attachment 12: Troubleshooting

18.0 Revision History

- Revision 11, Dated 03 March 2020
  - Annual review
  - Updated copyright information.

- Revision 10, Dated 31 January 2020
  - Added a graduated cylinder to Section 6.2.3
  - Updated standard information in Sections 7.2.3 - 7.2.5 and 7.2.9.2
  - Removed section 10.7.3 about soil dilution for Agilent 7500
  - Updated listed levels in Attachments 1, 4, 5, 7 and 11.

- Revision 9, Dated 30 November 2018
  - Annual review
  - Updated Section 10.6.3 to note the Tune must be rerun after any changes to the instrument.
  - Updated MDL procedure to reference SOP CA-S-Q-006.

- Revision 8, Dated 30 November 2017
  - Annual review
  - Updated Tuning criteria in Section 10.6.3 for Mass resolution
  - Added item 9 in Section 16 for mass resolution criteria

- Revision 7, Dated 30 April 2017
  - Annual Review
  - Removed all references to instrument 024
  - Updated Sections 7.2.1, 7.2.2, 7.2.7 and 7.2.9 to include the frequency to prepare

the standards
  - Updated Section 7.3.1 to reflect current definition of Reagent Water
  - Updated Section 9.2 to control common lab contaminates to the RL
  - Added Section 11.9 referencing Trivalent Chromium calculation
  - Added Sections 11.10 & 11.11 detailing data reporting and review
  - Added current Section 12.3 – detailing LOQVs
  - Updated Section 12.8 regarding Training Requirements/Responsibilities
  - Renumbered Section 12

- Revision 6, Dated 30 April 2016
  - Annual Review
  - Removed AFCEE references
  - Corrected Section references
  - Removed pre-2011 revision histories
  - Added MSA requirement
  - Added NOTE to Section 9.4 regarding TCLP MS/MSD failures
  - Added new Attachment 9 with TCLP regulatory limits

- Revision 5, Dated 30 April 2015
  - Annual Review
  - Language and formatting changes throughout
  - Corrected tuning sample requirement from 4 replicates to five replicates
  - Changed 12.4.2 to 12.5
  - Added Section 2.4
  - Added new Section 2.3 to describe reaction cell
  - Added new Section 4.2.3
  - Added batch definition
  - Deleted Attachment 2
  - Deleted Attachment 4
  - Added new Attachment 2 Recommended elemental equations
  - Section 4.3.2 enlarged expected IS intensities
  - Integrated Sections 4.4 and 4.5 into 4.2
  - Created new Section 4.3 Doubly charged ions
  - Added new Section 6.2.3 volumetric flasks
  - Section 7.0 added standard reagent language
  - Section 7.2.1.4 corrected Mg addition to standard
  - Section 7.2.2 replaced P/A section with same section from DV-MT-0025
  - Added P/A section to Attachment 7
  - Section 7.1.3 added reference to standards SOP
  - Added all new standard prep information into Sections 7.2.3 – 7.2.9
  - Section 9.3 changed spike level from midpoint of LR to midpoint of cal curve
  - Added note to Section 9.4
  - Changed timing of ICSA to beginning of analytical run and every 12 hours
  - Changed concentration limits for SD in Section 9.7 to 50x MDL
  - Added method modification 8 to address SD limit
  - 10.6.7 added DoD 5.0 language to corrective action
  - 10.7.5 added DoD requirement to dilute above daily LR
  - 11.7 changed to dry weight correction
  - 12.2.1 changed MDLV spike level to 2-4x MDL

- Revision 4, Dated 09 April 2014

- Annual Review
- Updated Section 7.2 to reference TALS for how to make standards
- Added Section 7.3.4 for 5%HNO3/5%HCL for Zr
- Added Maintenance Section 10.3
- Added Troubleshooting Section 10.4
- Added DoD QSM 5.0 criteria to Section 9 and 10
- Added Attachment 12 for troubleshooting

- Revision 3 Dated 15 July 2013
  - Annual Review
  - Corrected formatting
- Revision 2.1, Dated 13 July 2012
  - Updated Sections 9.1, 10.1, 10.2 and 12.1 to reflect current practice
  - Added Section 10.4.2 on Oxide/Doubly Charged controls
  - Updated Section 10.4.6 to control the calibration blanks to ½ the RL

- Revision 2, dated 2 December 2011
  - Removed references to Perkin Elmer instrument and added Agilent 7700 instrument.
  - Updated Section 9.1
  - Updated method blank acceptance criteria to meet requirements of DV-QA-003P.
  - Updated references
  - Updated Section 7.2.1 for the Agilent 7700
  - Updated Section 7.2.2 for the newer instruments
  - Added the Agilent 7700 daily linear range solution
  - Added Section 7.2.9 on how to make the internal standard
  - Updated Section 7.2.10 for both instruments
  - Split Section 7.3.2 diluent into two solutions based on the instrument
  - Revised Section 8 notes to include information in old Attachment 7 and removed this attachment.
  - Updated Sections 9.4 and 9.5 to control the LCS and MS/MSD to historical limits
  - Added preparation of the Post Digestion Spike to Section 10.5
  - Updated MDLV spike level to 1-3 times the MDL
  - Updated Section 12.3 to not digest the IDL solution.
  - Updated Section 12.4 to clarify use of Daily Linear Range Verification Standard.
  - Updated Section 12.6.2 to 4 distinct ICV's
  - Added new attachment 1 to list reporting limits, removed old Attachment 2, and Attachment 7 (info in Section 8) and renumbered attachments. Updated references to attachments to revised numbers.
  - Updated Attachments 5-7 and 10 (new numbers).
  - Removed (renumbered remaining attachments)
  - Removed attachment 13 and inserted Section 11.8 to describe data review process with reference to SOP DV-QA-0020 for the example checklist and details on the review process.
  - Formatting and grammatical changes throughout

Earlier revision histories have been archived and are available upon request.

Attachment 1

Standard Reporting Limits for Water and Soil

Element Name	Element Symbol	Water (ug/L)	Soil (ug/Kg)
Aluminum	Al	50	5,000
Antimony	Sb	2.0	200
Arsenic	As	5.0	600
Barium	Ba	1.0	200
Beryllium	Be	1.0	100
Cadmium	Cd	1.0	100
Chromium	Cr	2.0	200
Cobalt	Co	1.0	100
Copper	Cu	2.0	250
Iron	Fe	50	5,000
Lead	Pb	1.0	150
Manganese	Mn	1.0	250
Molybdenum	Mo	2.0	200
Nickel	Ni	2.0	150
Selenium	Se	5.0	500
Silver	Ag	5.0	100
Strontium	Sr	5	---
Thallium	Tl	1.0	100
Thorium	Th	5.0	200
Tin	Sn	10	2,500
Tungsten	W	5.0	---
Uranium	U	1.0	100
Vanadium	V	5.0	500
Zinc	Zn	10	1,000
Zirconium	Zr	0.5	---

Attachment 2

Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

Where M = Total ion count rate at the specified mass.

Attachment 3

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO	-	AgN	-	-	AgC	-
^{123}Sb	AgO	-	AgN	SrCl	ZrS	CdC	-
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	-
^{138}Ba	SnO	SbOH	-	-	-	-	-
^{137}Ba	SbO	SnOH	-	MoCl	-	-	-
^{136}Ba	SnO	SnOH	-	-	-	SnC	-
^{135}Ba	SnO	SnOH	-	MoCl	-	-	-
^{134}Ba	SnO	SnOH	SnN	MoCl	-	SnC	-
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	-
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	-
^{9}Be	-	-	-	-	-	-	-
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS	-	-
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	-
^{111}Cd	MoO	MoOH	MoN	GeCl	-	-	-
^{110}Cd	MoO, ZrO	-	MoN, ZrN	GeCl, AsCl	SeS	MoC	-
^{113}Cd	MoO	MoOH	-	SeCl, AsCl	-	-	-
^{116}Cd	MoO	-	-	-	-	-	-
^{106}Cd	ZrO	-	MoN, ZrN	-	GeS	MoC, ZrC	-
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	-
^{52}Cr	ArO	ClOH	-	-	-	ArC	-
^{53}Cr	ClO	ArOH	KN	NCl, OCl	-	KC	-
^{50}Cr	SO	-	ArN	-	SO	ArC	Mo^{++}
^{54}Cr	-	ClOH	ArN, CaN	-	-	CaC	-
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS, SO_2H	CrC	-
^{208}Pb	-	-	-	-	-	-	-
^{206}Pb	-	-	-	-	-	-	-
^{207}Pb	-	-	-	-	-	-	-
^{204}Pb	-	-	-	-	-	-	-
^{55}Mn	KO	ArOH	KN	-	NaS	CaC	Cd^{++}

Attachment 3 (cont.)

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO	-	-	-	-	-	-
^{200}Hg	WO	WOH	WN	-	-	-	-
^{199}Hg	WO	WOH	-	-	-	-	
^{201}Hg	-	WOH	-	-	-	-	-
^{198}Hg	WO	TaOH	WN	-	-	WC	-
^{204}Hg	-	-	-	-	-	-	-
^{196}Hg	-	-	WN	-	-	-	-
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	-
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	-
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	-
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	-
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS	-	-
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	-
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	-
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	-
^{107}Ag	ZrO	ZrOH	-	GeCl	AsS	MoC	-
^{109}Ag	-	MoOH	MoN	GeCl	SeS	MoC	-
^{205}Tl	-	-	-	-	-	-	-
^{203}Tl	-	WOH	-	-	-	-	-
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	-
^{50}V	SO	-	ArN	-	-	ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	-
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	-
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	-

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Attachment 4

Internal Standards (IS) and Corresponding Metals

IS	ICP-MS 077/078
^{6}Li	Be
Sc	Na, Mg, Al, K, Ca
Ge	V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se
In	Mo, Ag, Cd, Sn, Sb, Ba
Ho	Tl, Pb, Th, U, W

Attachment 5
Interference Check Sample Components and Concentrations

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	110.0
Ca	100.0	110.0
Fe	100.0	110.0
Mg	100.0	110.0
Na	100.0	110.0
P	100.0	100.0
K	100.0	110.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.1
Ti	2.0	2.0
As	0.0	0.1
Sb	0.0	0.1
Be	0.0	0.1
Ba	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Pb	0.0	0.1
Mn	0.0	0.1
Ni	0.0	0.1
Nb	0.0	0.2
Pd	0.0	0.1
Pt	0.0	0.1
Se	0.0	0.1
Tl	0.0	0.1
Th	0.0	0.1
Sn	0.0	0.1
Ag	0.0	0.1
U	0.0	0.1
V	0.0	0.1
W	0.0	0.1
Zn	0.0	0.1

Attachment 6
Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
93	Niobium
105	Palladium
195	Platinum
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"**205**", 203	Thallium
232	Thorium
192	Tungsten
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
139	Lanthanum
118	Tin
238	Uranium
35, 37	Chlorine
98, 96, 92, **97**, 94, "95"	Molybdenum
72	Germanium (IS)
165	Holmium (IS)
115	Indium (IS)
6	Lithium (6+) (IS)
45	Scandium (IS)

Attachment 7: Tuning Solution and P/A Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below is a suggested solution covering a typical mass calibration range. Instrument manufacturer recommendations should be followed for tuning solutions.

The P/A solution is used to monitor the correlation between the Pulse and Analog parts of the electron multiplier. This solution is prepared at different concentrations depending on the current instrument conditions. The parent standard concentration is shown below.

Element	Tuning Concentration (µg/L)	P/A Concentration (mg/L)
Al		5
As		20
Ba	10	5
Be	40	20
Bi		5
Cd		20
Ce	10	
Co	10	5
Cr		5
Cu		5
Ge		10
In	10	5
Ir		5
^{6}Li		5
Li	10	
Lu		5
Mg	20	10
Mn		5
Mo		10
Na		5
Ni		10
Pb	10	10
Pd		10
Rh	10	
Ru		10
Sb		10
Sc		5
Sn		10
Sr		5
Tb		2.5
Th		50
Ti		50
Tl	10	50
U	10	50
V		50
Y	10	2.5
Zn		20

Attachment 8

Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

Attachment 9

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5,000	4,000
Barium	1,000	100,000	12,000
Cadmium	100	1,000	1,100
Chromium	500	5,000	5,200
Lead	500	5,000	5,500
Selenium	100	1,000	3,000
Silver	500	5,000	1,050

Attachment 10

Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run.	90 - 110% recovery. 6020 IS, 30-120% rec.	Terminate analysis; correct the problem; recalibrate.
ICB	Immediately after each ICV	The result is < ½ RL. 6020 IS, 30-120% rec.	Terminate analysis; correct the problem; recalibrate.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	90 - 110% recovery. 6020 IS, 30-120% rec.	Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCB	Immediately following each CCV.	The result must be < ½ RL. 6020 IS, 30-120% rec.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and every 12 hours.	Internal standard recoveries within 30-120%	See Section 9.6
ICSAB	Immediately following each ICSA.	ICSAB recoveries for spiked elements within 20%. Internal standard recoveries for ICSA and AB within 30-120%.	See Section 9.6
Daily Linear Range Verification	As needed by program	90-110% recovery 6020 IS, 30-120% rec.	See Section 12.4.2

Attachment 10

Summary of Quality Control Requirements (continued)

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
Method Blank	One per lot of 20 field samples or fewer.	The result must be < ½ RL. Sample results greater than 10x the blank concentration or samples for which the contaminant is < ½ RL, do not require redigestion or reanalysis.	Re-run once. If > ½ RL, redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
Laboratory Control Sample	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.4 for additional requirements.
Matrix Spike/Matrix Spike Duplicate	One MS/MSD pair per lot of 20 field samples or fewer	Must be within laboratory control limits	See Section 9.5 for additional requirements.
Serial Dilution	One per batch of 20 field samples or fewer.	90 - 110% recovery	See Section 9.8 for additional requirements.
Post-Digestion Spike	One per batch of 20 field samples or fewer.	75-125% recovery	See Section 9.9.

Attachment 11

Calibration, Calibration Verification, and Spike Concentrations

Element	Initial Calibration (µg/L)	ICV (µg/L)	CCV (µg/L)	LCS (µg/L)	MS/MSD (µg/L)	Post Digestion Spike (ug/L)
Aluminum	2000	800	1000	400	400	20000
Antimony	100	40	50	40	40	200
Arsenic	100	40	50	40	40	200
Barium	100	40	50	40	40	200
Beryllium	100	40	50	40	40	200
Cadmium	100	40	50	40	40	200
Calcium	2000	800	1000	--	--	--
Chromium	100	40	50	40	40	200
Cobalt	100	40	50	40	40	200
Copper	100	40	50	40	40	200
Iron	2000	800	1000	400	400	20000
Lithium	200	80	100	40	40	200
Lead	100	40	50	40	40	200
Magnesium	2000	800	1000	--	--	--
Manganese	100	40	50	40	40	200
Molybdenum	100	40	50	40	40	200
Nickel	100	40	50	40	40	200
Selenium	100	40	50	40	40	200
Silver	100	40	50	40	40	50
Strontium	200	80	100	80	80	200
Thallium	100	40	50	40	40	200
Thorium	100	40	50	40	40	--
Tin	100	40	50	40	40	200
Tungsten	100	40	50	40	40	200
Uranium	100	40	50	40	40	200
Vanadium	100	40	50	40	40	200
Zinc	100	40	50	40	40	200
Zirconium	100	40	50	40	40	--

This procedure has been developed for twenty elements. Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

Attachment 12

ICP-MS Troubleshooting Guide

Problem	Possible Cause/ Solution
High Calibration Blanks	Inspect historical blank data to determine root cause Inspect, clean or replace torch Inspect, clean or replace pump tubing or sample tubing Inspect, clean or replace nebulizer Remake blank solution Recalibrate instrument
Instrument Drift	Make sure instrument has warmed properly Condition cones to aid stability Reslope to correct for changing cone conditions during run Stop run, clean cones and start over with a new calibration
Erratic Readings, High RSDs	Check nebulizer pressure Check sample flow around the pump, adjust tension on pump tubing to ensure smooth flow Check for clogs in the uptake tubing, nebulizer, or valve Clean or replace nebulizer
Low Sensitivity	Clean cones Adjust lens voltages Remove and clean lens, remove and clean or replace reaction cell
Bad Tune: Bad Mass Cal	Adjust lens voltages, remove and clean lens
Bad Tune: High Oxides	Inspect, clean, or replace torch, nebulizer, and spray chamber

Eurofins TestAmerica Denver

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ICP Analysis for Trace Elements by SW-846 Method 6010C/D

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1.0 Scope and Application

1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICPAES). This procedure references Methods 6010C and 6010D for hazardous waste (RCRA) testing.

1.2 The elements that can be determined by this procedure are listed in Attachment 1, together with the routine reporting limits. Additional elements may be analyzed under Method 6010C and 6010D provided that the method performance criteria presented in Section 12.0 are met.

1.3 The laboratory digests all water samples according to SOP DV-IP-0010.

1.4 Silver concentrations must be below 1.0 mg/L in aqueous sample digestates and 100 mg/kg in solid matrix sample digestates. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. Samples with silver concentrations exceeding these levels must be re-prepared and reanalyzed using a smaller sample amount.

1.5 The digestion procedure for soil samples is described in SOP DV-IP-0015.

1.6 State or client specific requirements may take precedence over this SOP for water analyses. Review special instructions for each project before starting work.

2.0 Summary of Method

2.1 The laboratory uses simultaneous ICPAES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by a charge injection device (CID). The photo-currents from the charge injection device (CID) are processed and controlled by a computer system.

2.3 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

2.4 Refer to the appropriate SOPs for details on sample preparation methods: DV-IP-0010 for aqueous samples and DV-IP-0015 for soil samples.

3.0 Definitions

3.1 Dual View ICP – an ICP equipped with both radial and axial viewing capabilities.

3.2 Dissolved Metals - Those elements which pass through a 0.45-µm membrane. The sample is acidified after filtration.

3.3 Potentially Dissolved Metals - Potentially dissolved metals is the concentration of metals in solution after acidifying the sample with nitric acid to pH < 2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-µm membrane filter. This definition is based on the Colorado surface water regulations.

3.4 Suspended Metals - Those elements which are retained by a 0.45-µm membrane.

3.5 Total Metals - The concentration determined on an unfiltered sample following vigorous digestion.

3.6 Total Recoverable Metals - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.7 Reporting Limit (RL) - The lowest concentration to which results are reported without qualification. Details concerning RLs are presented in Policy DV-QA-009P.

3.8 Reagent Water - Water with a resistivity of 1 Megohm-cm or greater. The Eurofins TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

3.9 Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

4.1.1 Overlap of a spectral line from another element.

4.1.2 Unresolved overlap of molecular band spectra.

4.1.3 Background contribution from continuous or recombination phenomena.

4.1.4 Stray light from the line emission of high concentration elements.

4.2 A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.3 Spectral Interferences

Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte signal. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.4 Physical Interferences

An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable (see Section 9.11), then dilution of the sample may be necessary to overcome the interferences.

4.5 Chemical Interferences

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed, can be minimized by buffering the sample, matrix matching, or standard addition procedures.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be

removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Thermo Fischer ICP 6500E Trace Analyzer, Agilent 5100 and 5110 are

currently used. Instruments with demonstrated equivalent performance can also be used

6.1.2 Radio Frequency Generator

6.1.3 Argon gas supply

6.1.4 Coolflow or appropriate water-cooling device.

6.1.5 Peristaltic Pump.

6.1.6 Autosampler.

6.2 Supplies

6.2.1 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.2 Class A volumetric flasks.

6.2.3 Autosampler tubes.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards used in calculations shall be entered into the TALS Reagent Module with all applicable information (e.g., components, concentrations, expiration, etc.).

7.2 Shelf-Life

7.2.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, a one-year expiration will be assigned by the laboratory.

7.2.2 The expiration date of intermediate concentration standards or working standards will be set at six months or less and cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.2.3 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be

replaced immediately.

7.3 Standards

7.3.1 Standards used for calibration and quality control purposes must be NIST traceable, where available. Multi-component custom blend standards must be verified against a second-source standard before they are first put into use (the only exception is standards purchased directly from NIST), as described in SOP DV-QA-0015. If the standard has been purchased previously it does not need to be verified, but the COA must be inspected to confirm that there have been no changes to the standard analyte levels.

7.3.2 Stock standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in the Eurofins TestAmerica LIMS (TALS).

7.3.3 Intermediate calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 4.4 of this procedure.

7.4 Reagent Blank / Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

Fill a 20-liter carboy with about 18 liters of reagent water. Slowly add 1 liter of concentrated nitric acid and 1 liter of concentrated hydrochloric acid. Adjust the total volume to 20 liters. Mix carefully. Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.5 Stock ICSA and ICSAB Standards

The following standards are purchased from commercial sources:

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
ICP ICSA Mix	Fe Al, Ca, Mg	2,000 5,000
ICSAB-1	Sb, Mo, Sn, Ti Si SiO2	100 1,000 2,140
ICSAB-2	Be Ag, As, Ba, Cd, Co, Cr, Cu,	50 100

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
	Mn, Ni, Pb, Se, Sr, Tl, V, Zn B, K, Na, P	1,000
1000 Bi	Bi	1,000
1000 Li	Li	1,000
1000 Zr	Zr	1,000
1000 S	S	10,000
1000 Th	Th	10,000

7.6 ICSA Working Standard (ICP ICSA)

A combined working ICSA standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSA and ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSA standard concentrations shown in Attachment 4.

7.7 ICSAB Working Standard

A combined working ICSAB standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICP ICSA Mix	25
ICSAB-1	2.5
ICSAB-2	2.5
1000 Bi	0.25
1000 Li	0.25
1000 Zr	0.25
10000 S	0.25
10000 Th	0.05

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSAB standard concentrations shown in Attachment 4.

7.8 Calibration Check Standard (S1, S2)

The two calibration check standards are the same as the working ICAL standards (ICP ICAL1A and ICP ICAL2A) described in Section 7.12.

7.9 Laboratory Control Sample (LCS) Stock Standards

The LCS stock standards are purchased from commercial sources. The stocks are custom-made standards purchased at ready-to-use concentrations as follows:

LCS Stock Standards	Elements	Concentration (mg/L)
Spike Mix #1	As, Ba, Se, Si, Sn, Tl Be, Cd, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sr, Ti, V	200 100
Spike Mix #2	Ca, K, Mg, Na Al, Fe	5,000 1,000
Spike Mix #3	P S U W	2,000 1,000 200 100
Spike Mix #4	Bi B, Th Zn, Zr	200 100 50
Spike Mix #4B	Ag	5
Spike Mix #5	Sb	100

The soil and water LCSs are prepared according to the instructions in SOPs DV-IP-10 and DV-IP-0015. Final concentrations are shown in Attachment 2.

7.10 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

The same LCS stock standards described in Section 7.9 are also used to prepare matrix spikes and matrix spike duplicates. Final concentrations are shown in Attachment 2.

7.11 Post Digestion Spike (PDS) Standards (Analyte Addition Spike Standards)

The custom standards tabulated below are purchased from a commercial source. Add 0.06 mL of each to 6 mL (100X) of digestate or dilution of digestate.

PDS Stock	Elements	Conc. (mg/L)
ICP PDS 1	Ag, Be, Cd, Co, Cr, Cu, Mn, Ni, Sr, V Ba, Li, Pb, As, Se, Th, Tl, Zn U Al, Fe P Ca, K, Mg, Na,	5 5 10 20 50 100 200 2,000
ICP PDS 2	Mo, Ti, Zr B, Sb, Sn Si (SiO_2)	5 10 500 (1,070)

7.12 Initial Calibration (ICAL) Standards

7.12.1 Stock Calibration Standards

The following stock solutions are purchased from commercial sources.

Stock Standard	Elements	Conc. (mg/L)
ICP Cal Std #1	Si As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V	1,000 100
ICP Cal Std #3	P S U W Al, Fe, Na, Th U	1,000 1,000 100 100 10,000 1,000
ICP Cal Std #4	K Na Mg Ca Fe Bi Ag, Al, B, Zn, Zr	10,000 5,000 4,000 1,000 500 200 100

7.12.2 Working Initial Calibration Standard (ICP ICAL1A)

Add 10.0 mL of ICP Cal Std #3 and ICP Cal Std #4 to a 1 liter volumetric flask partially filled with reagent blank solution. Add 2 mL of the As, Pb, Sb, Se, and Tl stocks. Dilute to the mark with reagent blank solution.

7.12.3 Working Initial Calibration Standard (ICP ICAL2A)

Add 10 mL of ICP Cal Std #3, 10 mL of the 10,000 mg/L Al and Fe stock standards, 50 mL of 10,000 mg/L Na stock, 1 mL of the 10,000 mg/L Th standard and 4 mL of the 1,000 mg/L U standard to a 1,000-mL volumetric flask partially filled with reagent blank and dilute to the mark with reagent blank.

7.13 Initial Calibration Verification (ICV)

7.13.1 ICV Stock Standards

The following stock solutions are purchased from commercial sources:

Stock Standard	Elements	Conc. (mg/L)
ICP CalStd1-SS	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V Si (SiO2)	100 1,000 (2,140)

Stock Standard	Elements	Conc. (mg/L)
ICP CalStd4-SS	Ag, Al, B, Zn, Zr	100
	Bi	200
	Ca	1,000
	Fe	500
	K	10,000
	Mg	4,000
	Na	5,000
ICP CalStd 3-SS	P	1,000
	S	1,000
	U	100
	W	100
Al-SS, Fe-SS, Na-SS, Th-SS Stocks	Al, Fe, Na, Th	10,000

7.13.2 Working High Initial Calibration Verification (ICP ICVH)

Add 0.40 mL of ICP CalStd3-SS stock standard, 0.40 mL of the 10,000 mg/L Al standard, 0.8 mL of the 10,000 mg/L Fe and Na standards and 0.030 mL of the 10,000 mg/L Th standard to a 100 mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

Note: For Method 6010D the ICV working solutions must be prepared daily.

7.13.3 Working Initial Calibration Verification (ICP ICV)

Add 0.25 mL of ICP CALStd1-SS and ICP CalStd4-SS to a 100-mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

Note: For Method 6010D the ICV working solutions must be prepared daily.

7.14 Reporting Limit Standard

7.14.1 RL Stock Standards

The low level ICV/CCV verification stock standards are custom-made commercial standards used to make the working RL standard:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP-LLCCV-5-REV1	K	300
	Na	100
	Ca, Mg	20
	Al, Bi, Fe	10
	U	6
	Ni	4
	Zn, Li, Se	2
	As, Cu, Th	1.5
	Tl	1.2
	Ba, Cr, Co, Mn, Ag, Sr	1
	V	1
	Pb	0.9
	Cd	0.5
	Be	0.1
ICP LLCCV-2A	P	300
	Si	50
	B, Sn, S	10
	Mo, Sb	2
	Zr, Ti	1

7.14.2 Reporting Limit Working Standard (ICP CRI)

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV-5-REV1	1
ICP-LLCCV-2A	1

Adjust to volume (100 mL) using the reagent blank solution. The working RL standard must be prepared daily.

7.15 High Continuing Calibration Verification (ICP CCVH)

Perform a 2x dilution of the working ICP ICAL2A solution (Section 7.12.3) with reagent blank solution.

7.16 Continuing Calibration Verification (ICP CCV)

Perform a 2x dilution of the working ICP ICAL1A solution (Section 7.12.2) with reagent blank solution.

7.17 Low Level ICV/Low Level CCV (ICP LLCCV)

The low level ICV/CCV verification stock standards are custom-made commercial standards as follows:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP LLCCV-4	K	300
	Na	100
	Ca, Mg	20
	Al, Bi, Fe	10
	U	6
	Ni	4
	Li, Se, Zn	2
	As, Cu, Tl, Th	1.5
	Ba, Cr, Co, Mn, Ag, Sr, V	1
	Pb	0.9
	Cd	0.5
	Be	0.1
ICP LLCCV-2	P	300
	Si	50
	B, S, Sn	10
	Mo, Sb	2
	Zr, Ti	1.0

7.17.1 Low Level ICV \ Low Level CCV, Working Standards

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV-4	1
ICP-LLCCV-2	1

Adjust to volume (100 mL) using the reagent blank solution.

7.18 Linear Range Verification Standard (LR)

The LRA standard is prepared from single element stock standards of each metal obtained from a commercial source. The stock standards are each purchased at a concentration of 1,000 mg/L except for Iron and Silicon which are each at a concentration of 10,000 mg/L. The LRA is prepared by taking the appropriate volume of each stock and adding it to a 500 mL volumetric flask partially filled with reagent blank and diluted to the mark after all elements have been added. The volume of each stock solution of each metal and volume used, along with final concentrations of each are listed in the following table.

Elements	Stock Conc. (mg/L)	Volume of stock (mL)	Final Conc. (mg/L)
Cd,	1,000	1.0	2
Co, Mo, Se, Tl	1,000	2.5	5
As, B, Cr, Cu, Mn, Ni, Pb, Sr, Ti, V, Zn	1,000	5.0	10
Ba	1,000	6.0	12
Fe	10,000	25	500
Si (SiO_2)	10,000	2.5	50 (107)

7.19 Internal Standard (ICP IS)

7.19.1 For the Agilent 5100 and 5110 add 0.5 ml of 10,000 mg/L Yttrium to 1000 ml of reagent blank.

7.19.2 For the Thermo 6500 add 0.25 ml of 10,000 mg/l Yttrium to 1000 ml of reagent blank.

7.20 Reagents

7.20.1 Concentrated nitric acid (HNO_3), trace metals grade or better.

7.20.2 Concentrated hydrochloric acid (HCl), trace metals grade or better.

7.20.3 Reagent water must be produced by a Millipore DI system or equivalent, with a minimum resistivity of 1.0 Mohm/cm at 25 ˚C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation [1]	Holding Time [2]	Reference
Waters	HDPE	50 mL	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool ≤ 6 °C [3]	180 Days	N/A

[1] Aqueous samples are preserved with nitric acid to a pH of < 2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required for most programs. Preservation must be verified prior to analysis.

[2] Inclusive of digestion and analysis.

[3] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot volume for both analyses must be refrigerated.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. DoD QSM 5.0 or 5.1 QC Acceptance Criteria for ICP analyses are presented in Attachment 11. The criteria must be met unless otherwise documented in the project documents.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.3 Method Blank

The blank is de-ionized water taken through the procedure as if it were a sample. For soil samples analyzed under the DoD QAPPs, the method blank consists of < 1 mm glass beads that have been processed in the same manner as the samples. A method blank is required with every batch of 20 or less samples.

Acceptance Criteria: The method blank must not contain any analyte of interest above ½ the reporting limit or above one-tenth of the

concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.4 Laboratory Control Sample (LCS)

The LCS is prepared as described in Section 7.9. One LCS is required with each analytical batch.

Acceptance Criteria: The recovery of the LCS must be within historical control limits. Historical control limits are based on three standard deviations of past results, and must be 80 - 120% or tighter. In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative. The process of establishing control limits is described in more detail in the Policy DV-QA-003P. The control limits are stored in TALS.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS/MSDs are prepared as described in Section 7.10. One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require the MS/MSDs to be run at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DoD QSM 5.0 or 5.1 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in

order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client’s needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.6 Method of Standard Additions (MSA)

9.6.1 This technique involves constructing a calibration curve in the sample matrix itself to compensate for any sample interferents that may enhance or depress 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. Attachment 8 provides more guidance on performing MSA analyses.

9.6.2 EPA Method 1311 (Section 8.4) requires that the MSA be used as the calibration method if the MS or MSD recoveries for TCLP extracts are less than 50% and the sample result is within 80 - 100% of the regulatory level. Attachment 4 provides a list of the regulatory limits. Although the MSA calibration technique may be used with only the sample and a single spiked point, Method 1311 specifies that three spiked points must be used along with the sample.

9.6.3 TALS does not currently have the capability to report results from an MSA-based analysis. If an MSA must be performed, the sample results must be calculated using the MSA spreadsheet (stored at R:\QA\Edit\Forms\Metals\MSA Worksheet - Water) and reported in an NCM. All of the associated samples must then be recalculated against the MSA spreadsheet. The completed spreadsheet must be saved and attached to the analytical batch in TALS along with the raw data.

9.6.4 A manual “N’ flag must also be added to all of the affected samples in TALS, indicating the presumptive evidence for the analyte. This flag

signals the Project Manager and indicates that narration of the result is required.

9.7 Serial Dilution Test

A dilution test is performed for each batch of samples. The purpose of this test is to ensure that neither positive nor negative interferences are biasing the analytical results. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1:5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination. For DoD QSM 5.0 or 5.1 the serial dilution is required if the MS or MSD fails and the parent concentration is greater than 50x the LOQ prior to dilution. 6010D requires the parent sample to be at least 25x higher than the RL to be calculable and sets the recovery limit at 20%.

Corrective Action: If the two results do not agree within the required limits, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and the failure is addressed in the case narrative to alert the client that a matrix affect may be present. For DoD QSM 5.0 or 5.1 a J-flag is added to the parent sample for the specific analyte if the acceptance criteria are not met.

9.8 Post Digestion Spike (PDS)

Whenever the MS/MSD recoveries are unacceptable, a PDS spike must be performed. The PDS spike is prepared as described in Section 7.10. Some programs require a PDS analysis whenever the serial dilution test fails. Other programs (e.g., DoD QSM 5.0 or 5.1) require a PDS to be included in every batch. Check project requirements. For programs where a PDS is required, the same sample that was used for the serial dilution test should be used for the PDS.

Acceptance Criteria: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% for Method 6010C and 75 - 125% for 6010D.

Corrective Action: If the spike is not recovered within the specified limits, a matrix effect is confirmed. For DoD QSM 5.0 or 5.1 a J-flag is added to the parent sample if the sample concentration is less than 50x the LOQ prior to dilution. Any failures are flagged and should be described in the report case narrative.

9.9 Interference Check Analysis (ICSA / ICSAB)

The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Sections 7.5, 7.6, and 7.7 for the preparation of the ICSA and ICSAB solutions. Attachment 4 lists the final concentrations. All analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run.

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements must be ≤ 2x RL. The DoD and AFCEE programs have their own criteria based on the version used. For DoD QSM 5.0 or 5.1 the non-spiked analytes must be less than the absolute value of the LOD unless they are verified impurities. For 6010D the non-spiked analytes must be less than the absolute value of the RL.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows: If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted. The sample data may also be accepted if the affected element was not required. If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2x RL, then the field sample data can be accepted. If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2x RL, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA. If the data do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed.

9.10 Monitoring Internal Standard Results

Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

Acceptance Criteria: If the internal standard counts fall within ± 30% of the counts observed in the ICAL blank, then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the field samples must be diluted and reanalyzed;

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0010 and DV-IP-0015).

10.4 Calibration

10.4.1 Instrument Start Up

Set up the instrument with the operating parameters recommended by the manufacturer. Complete any required preventative maintenance and record in the ICPAES Preventative Maintenance Log. Preventive maintenance recommendations are listed in the Eurofins TestAmerica Denver Quality Assurance Manual. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.2 Initial Calibration (ICAL)

The calibration curve is established on each day of operation using a blank and one standard. The preparation of the ICAL standards is described in Section 7. The final concentrations of the ICAL standards are presented in Attachment 6. The validity of the calibration curve is confirmed by analysis of the ICV, CCV, ICB, RL Check standard and Low Level ICV/CCV) which are run immediately after the ICAL. Some programs also require a high-level verification check (see Section 10.4.9).

10.4.3 Initial Calibration Verification (ICP ICVH and ICP ICV)

Calibration accuracy is verified using a second-source standard (ICP ICVH and ICP ICV) that is at or below a concentration near the mid-point of the working range. The ICV is analyzed immediately after the ICAL. The preparation of this standard is described in Section 7. The concentration of the ICV standard is presented in Attachment 6.

Acceptance Criteria: The ICV result must fall within 10% of the true value for that solution. The relative standard deviation must be < 5% (the laboratory is using at least two exposures for all ICP analyses).

Corrective Action: If the ICV fails to meet acceptance limits, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.4 Mid Level Continuing Calibration Verification (CCV)

The preparation of the CCV solution is described in Section 7. The final concentrations of the CCVs are presented in Attachment 6. Note that the CCV is made at a different concentration than the ICV to meet NELAC requirements. CCVs are analyzed after the ICV, after every ten samples, and at the end of the analytical run.

Acceptance Criteria: The CCV must be within 10% of the expected value. The relative standard deviation must be < 5%.

Corrective Action: If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue*. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed.

10.4.5 6010C - Low Level Initial Calibration (LLICV) and Continuing Calibration Verification (LLCCV)

The preparation of the LLCCV solution is described in Section 7. The low-level CCV needs to be analyzed at the beginning and end of every run sequence. If low level samples are expected then the low-level CCV should also be run every ten samples.

Acceptance Criteria: The LLCCV must be within +/-30% of the expected value to meet Method 6010C requirements.

Corrective Action: If the LLCCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue*. If the calibration cannot be verified within theses specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the LLCCV standard successfully analyzed. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed. Eurofins TestAmerica will not hold samples with concentrations greater than 10x the reporting limit to the 30% acceptance criteria.

10.4.6 Initial Calibration Blank (ICB)

System cleanliness is verified by analyzing an ICB after the first CCV. The preparation of the ICB is described in Section 7.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD QSM 5.0 or 5.1 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the ICB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.7 RL Calibration Check Standard (ICP CRI)

Calibration accuracy at the RL is verified by analyzing a standard prepared at a concentration at or below the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. Alternate RL standard concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents Module in TALS.

Acceptance Criteria: For routine work the acceptance limits are ± 50% of the expected value. For **6010D and DoD QSM** the acceptance limits are ± 20%.

Corrective Action: If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.8 Lower Limit of Quantitation Check (LLQC)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the LLICV/CCV is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliquots spiked at the LLOQ.

Acceptance Criteria: LLQC is verified when all analytes are detected within ± 30% of their true value with an RSD ≤ 20%.

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.9 High-Level Calibration Check Standard

The method 6010C defines the linear working range used for daily analysis based on the LDR studies performed every six months, in which case this standard is not required. However, some programs require verification of the high end of the linear range at different frequencies. The DoD QSM 5.0 OR 5.1 requires that the linear range must be verified on a daily basis. For DoD QSM 5.0 or 5.1 and Method 6010D samples, the spike level of the highest standard analyzed defines the linear range for that day.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the High-Level Calibration Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met for this calibration or the sample

results cannot exceed the level of the highest calibration standard.

10.4.10 Continuing Calibration Blank (CCB)

CCBs, prepared as in Section 7.4, are analyzed after each CCV.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD. Method 6010D sets the CCB upper limit at the RL.

Corrective Action: If the CCB is greater than these limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, instrument maintenance should be considered, the calibration re-verified, and all samples analyzed since the last successful CCB must be reanalyzed.

10.5 Sample Analysis

10.5.1 Replicate Readings

The laboratory averages the results from two exposures for Axial and Dual View ICP for each standard, field sample, and QC sample due to sample volume limitations of the autosampler tube.

10.5.2 Rinse Time between Samples

Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless, following the protocol outlined in 12.7, it can be demonstrated that a shorter rinse time may be used.

10.5.3 The following analytical sequence is used:

Instrument Calibration
High Standard Verification
ICV
LLICV (6010C only)
CCV
ICB
RL Verification Standard
LLQC (as needed)
ICSA
ICSAB
LRA
CCV

CCB
LLCCV (6010C only)
10 samples
CCV
CCB
LLCCV (6010C)
10 samples
CCV
CCB
LLCCV (6010C)
Repeat sequence with 10 samples between CCV/CCB pairs
CCV
CCB
LLCCV (6010C)

10.5.4 Full method-required QC must be available for each wavelength used in determining reported analyte results. Guidelines are provided in the appendices for minimizing contamination of samples and standards (Attachment 10) and troubleshooting (Attachment 9).

10.5.5 Dilutions for High Levels of Elements of Interest

For 6010, results must fall within the linear range. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid concentration.

10.5.6 6010D Mid-Run Recalibration

During the course of an analytical run, the instrument may be recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed.

10.5.7 Dilutions for High Levels of Interfering Elements

Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted until the interferent is at or below the working range. An NCM will be written in these instances.

10.6 Instrument Maintenance

See Section 20 in the QAM.

10.7 Troubleshooting

See Attachment 9.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves & Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 The procedure for performing the calculation of ferric iron is detailed in the Work Instruction WI-DV-0092, *Calculation Methods*.

11.3 ICV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}}\right) \times 100\%$$

11.4 CCV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}}\right) \times 100\%$$

11.5 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{SSR - SR}{SA}\right) \times 100\%$$

Where:

SSR	=	Spike Sample Result
SR	=	Sample Result
SA	=	Spike Added

The relative percent difference (RPD) of a matrix spike/matrix spike duplicate pair is calculated according to the following equation:

$$RPD = \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right] \times 100$$

Where:

MS	=	determined spiked sample concentration
MSD	=	determined matrix spike duplicate concentration

11.6 The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
$V1$	=	Final volume in liters after sample preparation
$V2$	=	Initial volume of sample digested in liters

11.7 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V	=	Final volume in liters after sample preparation
W	=	Weight in kg of wet sample digested
S	=	Percent solids/100

NOTE: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

11.8 The LCS percent recovery is calculated according to the following equation:

$$\%R = \left(\frac{\text{LCS Found Value}}{\text{LCS True Value}}\right) \times 100\%$$

11.9 The IEC's are calculated according to the following equation:

$$IEC = \left(\frac{\text{observed concentration}}{\text{observed concentration of the interfering element}}\right)$$

11.10 The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I	=	Sample result (Instrument reading)
S	=	Dilution test result (Instrument reading × 5)

Appropriate factors must be applied to sample values if dilutions are performed.

11.11 Documentation and Record Management

11.11.1 All sample data is uploaded to TALS. All sample preparation and analytical batch information, including the batch number(s), list of samples, preparation analyst and date, instrument analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes) is recorded in TALS.

11.11.2 Raw data is scanned or saved directly as a PDF and is attached to the analytical batch in TALS.

11.12 Reporting

11.12.1 Reporting units are µg/L for water samples and mg/kg for solid samples.

11.12.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

11.12.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.12.4 All associated data are entered or uploaded into the LIMS as required.

NOTE: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.12.5 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. See SOP DV-QA-0020 for more detail on the review process. Any manually transcribed data must be reviewed in its entirety by the second level data reviewer.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly. DoD QSM 5.0 or 5.1 requires the MDLV spike level to be 2 - 4 times the calculated MDL.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Instrument Detection Limit Study

12.3.1 Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each wavelength used for analysis.

12.3.2 Run seven blanks on three non-consecutive days.

12.3.3 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.4 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.3.5 For Method 6010D the IDL solutions:

- Should be prepared with each of the different matrices analyzed on the instrument;
- Should be prepared with 10 replicates for each matrix;
- Should have all the replicates for each matrix analyzed in a single day.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards is required for the initial and on-going studies, and one of the levels must be close to the upper end of the range. The highest concentration must be within 10% of the stated concentration.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions. Certain programs do not allow the use of LDRs for reporting purposes and instead require all sample results to fall below the highest daily standard analyzed.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Background Correction Points

12.5.1 To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength of interest and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations.

12.5.2 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

12.6 Interelement Corrections (IECs)

12.6.1 ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

12.6.2 When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

12.6.3 Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. Note that the USACE program requires a control limit of 2x |MDL|, which is feasible when verified MDLs are used.

12.6.4 To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element." Method 6010D requires that the IEC standards include Al, B, Ba, Ca, Cu, Fe, Mg, Mn, Mo, Na, Ni, Se, Si, Sn, V, and Zn.

12.6.5 Dual-View ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the CID detector instruments as reflected by the ICSA response.

12.7 Rinse Time Determination

12.7.1 Rinse times must be determined annually.

12.7.2 To determine the appropriate rinse time for a particular ICP system, a standard containing the highest concentration level that would be reported for samples is aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system.

12.7.3 For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level).

12.7.4 Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

12.7.5 The ICP instruments use an intelligent rinse program. The intelligent rinse lengthens the rinse time whenever a sample result for a known problem analyte is above a set concentration.

12.8 Demonstration of Capabilities

12.8.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

12.8.2 IDOCs and on-going proficiency demonstrations are conducted as follows: Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

12.8.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.9 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

13.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with federal, state, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Acid solutions from ICP drain - Waste Stream J

14.2.2 Metals waste potentially contaminated with Cat 1 radioactive materials – Waste Stream RJ

Note: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition and all promulgated updates, EPA Office of Solid Waste, through January 2008.

15.1.1 Method 6010C, Revision 3, Update IV, February 2007.

15.1.2 Method 6010D, Revision 4, Update V, July 2014.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

15.4 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.1, 2017.

15.5 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.2, 2018.

15.6 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories Version 5.3, 2019.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6010C	This procedure uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.
2	EPA 6010C	The alternate run sequence presented in Section 10.5.3 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.
3	EPA 6010C	Method 6010 states that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific "concentration range around the calibration blank." Because of the lack of definition for "concentration range around the calibration blank," the laboratory has adopted the procedure in EPA CLP ILMO4.0 for determining IECs,
4	EPA 6010C	Section 9.9 of Method 6010C states: "If less than acceptable accuracy and precision data are generated, additional quality control tests are recommended prior to reporting concentration data for the elements in this method." The dilution test helps determine if a chemical or physical interference exists. Because the laboratory sometimes does not have prior knowledge if the MS/MSD will be within criteria, the analyst may select to perform a dilution test on one sample in each preparation batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. In this procedure, matrix interference is determined by evaluating data for the LCS, MS/MSD, and serial dilutions. The laboratory must request documented, clear guidance when an unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.0 Attachments

Attachment 1 Metals Analyzed by ICP and Reporting Limits
Attachment 2 Matrix Spike and Aqueous Laboratory Control Sample Levels
Attachment 3 Low Level ICV and CCV Spiking Levels
Attachment 4 Interference Check Sample Concentrations
Attachment 5 TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
Attachment 6 6500 Initial Calibration &Continuing Calibration Verification Standards
Attachment 7 Summary of Quality Control Requirements
Attachment 8 MSA Guidance
Attachment 9 Troubleshooting Guide
Attachment 10 Contamination Controls
Attachment 11 DoD QSM 5.0 or 5.1 QC Acceptance Criteria

18.0 Revision History

Revision 9, dated 20 November 2020
- Annual review
- Added Agilent 5100 and 5110 to instrument list in Section 6.1.1
- Added Section 7.19 on how to make the internal standard.

Revision 8, dated 31 October 2019
- Annual review
- Minor formatting and language changes throughout
- Updated Standard ID's and Concentrations in Section 7.0
- Updated Concentrations in Attachments #1 thru #6

Revision 7, dated 31 July 2018
- Annual review
- Added Sections 7.14.1 and 2 to use the ICVL/CCVL to make the RL Standard
- Added Silicon, Silica, and Boron to the list of elements in Attachment 6.

Revision 6, dated 31 July 2017
- Annual review
- Added reference to QSM 5.1 throughout SOP where applicable
- Updated spiking amounts in Section 7.12.2 to be based off 1L instead of 500ml
- Added LCSD requirement to Section 9.5 for DoD when not enough volume for MS/MSD
- Added Section 11.12 regarding reporting requirements
- Added current Section 12.2 regarding LOQVs

Revision 5, dated 31 July 2016
- Annual review
- Minor formatting and language corrections throughout
- Removed references to AFCEE and USACE
- Added Section 3.8 reagent water definition
- Added six-month expiration for intermediate standards in Section 7.2.2
- Added clarification to Section 7.3.2 regarding new standard verification
- Added new Section 7.18 explaining daily LR standard
- Removed cooling requirement for water samples in Section 8.0
- Added information to MS/MSD Section 9.5 to reflect current policy
- Changed Section 9.9 to Section 9.6, renumbered other sections accordingly
- Added information to Section 9.6 regarding the MSA requirement for TCLP extract samples
- Created subsections 9.6.1 - 9.6.4
- Added language to Section 10.4.9 to clarify daily linear range requirements
- Added Section 11.2 referencing the ferric iron work instruction
- Added requirement to review all manually transcribed data at second level review (Section 11.11.3)
- Archived pre-2011 revision histories

Revision 4, dated 31 December 2015
- Added requirements for Method 6010D to the SOP
- Minor grammar and formatting corrections throughout
- Added list of IEC test analytes to Section 12.5.4
- Added Section 10.5.6 regarding mid-run recalibration

- Added Section 12.2.5 defining 6010D IDL studies

Revision 3, dated 31 July 2015
- Annual review
- Updated Section 7.4 for how to make the 5% HNO_3/5% HCl solution
- Updated Section 12.2 for MDLV spike level to 2-4x MDL
- Updated Section 12.8.2 to use LCSs instead of ICVs for the DOC
- Reformatting throughout
- Removed reference to silica holding time
- Added Maintenance and troubleshooting sections
- Replaced Section 11.10 to match current practice
- Removed Section 12.2
- Removed Sections 1.3.1 and 1.3.2
- Added new Section 1.6
- Removed reference to glass beads in Section 6.2
- Corrected Reagent and Standard formulae throughout to agree with current practice

Revision 2, dated 31 July 31 2014
- Annual review
- Updated Section 6.1.3 to specify purity of argon gas
- Added statement to section 9.1.2 to reference DoD QSM 5.0 criteria in Attachment 11
- Removed references to preparation of oil/oily samples throughout the document as the lab no longer supports this digestion method
- Added references for prep methods to section 15
- Added DOD QSM 5.0 QC acceptance criteria as Attachment 11

Revision 1, dated 15 July 2013
- Annual review
- Removed section 1.7
- Added section 3.8
- Corrected formatting
- Added section 11.12
- Removed Attachment 8, renumbered attachments and fixed references to attachments throughout the document

Revision 0.3, dated 13 July 2012
- Annual Review
- Clarified soil preservation for ICP only analysis, Section 8
- Updated section 9.1, 10.1, 10.2, and 12.1 to reflect current practice
- Updated sections 10.4.6 and 10.4.10 to control calibration blanks to ½ the RL

Earlier revision histories have been archived and are available upon request.

Attachment 1

Metals Analyzed by ICP and Reporting Limits

Element	Symbol	CAS #	6010 Analyte	Reporting Limit (µg/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	100	10
Antimony[trace]	Sb	7440-36-0	X	20	1.5
Arsenic[trace]	As	7440-38-2	X	15	2
Barium	Ba	7440-39-3	X	10	1
Beryllium	Be	7440-41-7	X	1	0.5
Bismuth	Bi	7440-69-9		100	10
Boron	B	7440-42-8	X	100	10
Cadmium[trace]	Cd	7440-43-9	X	5	0.5
Calcium	Ca	7440-70-2	X	200	50
Chromium	Cr	7440-47-3	X	10	1.5
Cobalt	Co	7440-48-4	X	10	1
Copper	Cu	7440-50-8	X	15	2
Iron	Fe	7439-89-6	X	100	20
Lead[trace]	Pb	7439-92-1	X	9	0.9
Lithium	Li	7439-93-2	X	20	5
Magnesium	Mg	7439-95-4	X	200	20
Manganese	Mn	7439-96-5	X	10	1
Molybdenum	Mo	7439-98-7	X	20	2
Nickel	Ni	7440-02-0	X	40	4
Phosphorus	P	7723-14-0	X	3,000	50
Potassium	K	7440-09-7	X	3,000	300
Selenium[trace]	Se	7782-49-2	X	20	1.8
Silicon	Si	7631-86-9		500	50
Silver[trace]	Ag	7440-22-4	X	10	1
Sodium	Na	7440-23-5	X	1,000	500
Strontium	Sr	7440-24-6	X	10	1
Sulfur	S	7704-34-9	X	100	15
Thallium[trace]	Tl	7440-28-0	X	15	1.3
Thorium	Th	7440-29-1		20	15
Tin	Sn	7440-31-5	X	100	10
Titanium	Ti	7440-32-6	X	10	1
Uranium	U	7440-61-1		60	20
Vanadium	V	7440-62-2	X	10	2
Zinc	Zn	7440-66-6	X	20	3
Zirconium	Zr	7440-67-7		15	3

Attachment 2

Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	10,000	10,000
Antimony	1,000	1,000
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	1,000	1,000
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	1,000	1,000
Calcium	50,000	50,000
Chromium	1,000	1,000
Cobalt	1,000	1,000
Copper	1,000	1,000
Iron	10,000	10,000
Lead	1,000	1,000
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	1,000	1,000
Molybdenum	1,000	1,000
Nickel	1,000	1,000
Phosphorous	20,000	20,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	2,000	2,000
Si (as SiO_2)	4,280	4,280
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Sulfur	10,000	10,000
Thallium	2,000	2,000
Thorium	1,000	1,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	1,000	1,000
Zinc	500	500
Zirconium	500	500

Attachment 3

Low Level ICV/CCV

Element	Level (µg/L)
Aluminum	100
Antimony	20
Arsenic	15
Barium	10
Beryllium	1
Bismuth	100
Boron	100
Cadmium	5
Calcium	200
Chromium	10
Cobalt	10
Copper	15
Iron	100
Lead	9
Lithium	20
Magnesium	200
Manganese	10
Molybdenum	20
Nickel	40
Phosphorous	3,000
Potassium	3,000
Selenium	20
Silicon	500
Si (as SiO_2)	1,070
Silver	10
Sodium	1,000
Strontium	10
Thallium	12
Thorium	15
Tin	100
Titanium	10
Uranium	60
Vanadium	10
Zinc	20
Zirconium	10

Attachment 4

Interference Check Sample Concentrations

Element	ICSA (µg/L)	ICSAB (µg/L)
Aluminum	500,000	500,000
Antimony	-	1,000
Arsenic	-	1,000
Barium	-	1,000
Beryllium	-	500
Bismuth	-	1,000
Boron	-	10,000
Cadmium	-	1,000
Calcium	500,000	500,000
Chromium	-	1,000
Cobalt	-	1,000
Copper	-	1,000
Iron	200,000	200,000
Lead	-	1,000
Lithium	-	1,000
Magnesium	500,000	500,000
Manganese	-	1,000
Molybdenum	-	1,000
Nickel	-	1,000
Phosphorous	-	2,000
Potassium	-	10,000
Selenium	-	1,000
Silicon	-	10,000
Silica	-	21,400
Silver	-	1,000
Sodium	-	10,000
Strontium	-	1,000
Sulfur	-	10,000
Thallium	-	1,000
Titanium	-	1,000
Vanadium	-	1,000

Attachment 4

Interference Check Sample Concentrations (cont'd)

Element	ICSA (µg/L)	ICSAB (µg/L)
Tin	-	1,000
Thorium	-	2,000
Uranium	2,000	2,000
Zinc	-	1,000
Zirconium	-	1,000

Attachment 5

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

Element	Reporting Level (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5,000	5,000
Barium	10,000	100,000	12,000
Cadmium	100	1,000	2,000
Chromium	500	5,000	6,000
Lead	500	5,000	6,000
Selenium	250	1,000	3,000
Silver	500	5,000	1,050
Copper	100	N/A	3,000
Zinc	200	N/A	500

Attachment 6

6000 Dual View Calibration, ICV & CCV Standards

Element	Calibration Level	ICV (µg/L)	CCV (µg/L)
Aluminum Lo	1,000	250	500
Aluminum Hi	100,000	40,000	50,000
Antimony	1,000	250	500
Arsenic	1,000	250	500
Barium	1,000	250	500
Beryllium	1,000	250	500
Bismuth	2,000	500	1,000
Boron	1,000	250	500
Cadmium	1,000	250	500
Calcium	10,000	2,000	5,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron Lo	5,000	1,250	2,500
Iron Hi	100,000	80,000	50,000
Lead	1,000	250	500
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Phosphorous	10,000	4,000	5,000
Potassium	100,000	25,000	50,000
Selenium	1,000	250	500
Silicon	10,000	2,500	5,000
Si (as SiO2)	21,400	5,350	10,700
Silver	1,000	250	500
Sodium Lo	50,000	12,500	25,000
Sodium Hi	500,000	80,000	250,000
Strontium	1,000	250	500
Sulfur	10,000	4,000	5,000
Thallium	1,000	250	500
Thorium	10,000	3,000	5,000
Tin	1,000	500	500
Vanadium	1,000	250	500
Uranium	5,000	-	2,500
Zinc	1,000	250	500
Zirconium	1,000	250	500

Attachment 7
Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤ 5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90 - 110% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
RL Standard	At the beginning of the run	Results must within 50%	Terminate analysis; Correct the problem; Recalibrate.
LLICV/CCV	At the beginning of the run and after every 10 samples	Recovery must be within 30%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable LLCCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- ½ RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10	See Section 9.10
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10
Dilution Test	One per prep batch.	For samples > 10x LOD (after dilution)' dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.

See Section 10.5.3 for run sequence to be followed.

Attachment 7

Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to ½ the RL. Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < ½ RL may not require redigestion or reanalysis (see Section 9.3)	Re-run once in a clean tube. If > ½ RL, re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.4)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see 9.5)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits.	See Corrective Action for Matrix Spike.

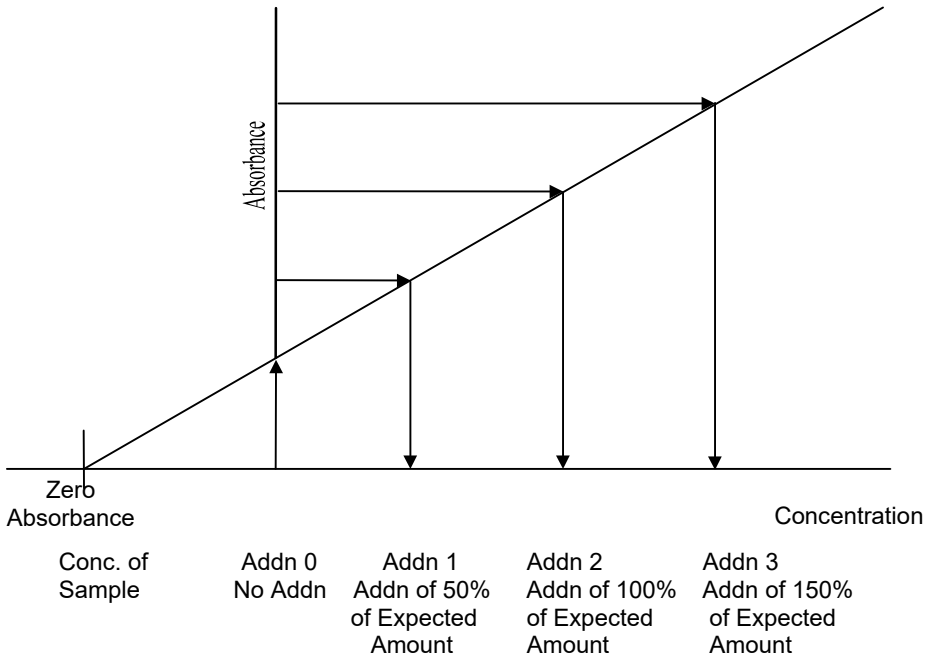
Attachment 8

MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear (r=0.995 or greater) over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 9

Troubleshooting Guide

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Replace RF generator
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

Attachment 10

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves should not be used in the metals laboratory because the powder contains silica and zinc as well as other metallic analytes.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Linear Dynamic Range (LDR) or high-level standard check	Run an LDR or high-level check standard at least once every 6 months. When calibrating with a single standard and a blank, the daily LDR standard must be analyzed at a concentration greater than any samples analyzed that day. Data cannot be reported above the high calibration range without an established/passing high-level check standard. Must be within ± 10% of expected value. Dilute samples within the calibration range or re-establish/verify the LDR.
Initial Calibration (ICAL)	Measure a minimum of one high standard and a calibration blank, daily. If more than one standard used, then $r^2 \geq 0.99$ ($r \geq 0.995$), otherwise no acceptance criteria. The ICAL must pass before running any samples. NOTE: The laboratory currently performs duplicate burns for the ICPAES method.
Initial Calibration Verification (ICV)	Run second-source standard once after each ICAL and prior to sample analysis. All reported analytes must be within ± 10% of expected value. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV after every 10 field samples, and at the end of the analysis sequence. All reported values within ± 10% of expected value If the CCV is above the project acceptance limits and there are no detections in the samples, Eurofins TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV. Or Immediately (within one hour) 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 analytes(s) in all samples since the last acceptable CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-Level Calibration Check Standard (Low-level ICV)	Run low-level standard at a concentration ≤ LOQ daily after one-point ICAL. All reported analytes must be within ± 20% of expected value. Correct any problems, then reanalyze or repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP
(continued)

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Initial and Continuing Calibration Blank (ICB.CCB)	Analyze calibration blank before analyzing samples, after every 10 field samples, and at the end of the analysis sequence. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. (13ICP) If not accepted by client, ICB/CCB must be <LOD. If criteria not met, correct problem If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed. Correct any problems and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCB failures due to carryover may not require an ICAL.
Interference Check Solution (ICS)	Run the ICS at the beginning of an analytical run (after ICAL and prior to sample analysis). ICS-A: Absolute value of concentration for all non-spiked analytes must be < LOD (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of expected value. (Note: ICS-AB not needed if instrument can read negative responses.) Correct any problems and reanalyze ICS. Do not analyze samples without a valid ICS. NOTE: TAL Denver has a letter from the ICSA standards manufacturer for many of the elements.
Method Blank	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common lab contaminants not detected > LOQ. (2CLC) For ICP, common lab contaminants are: Al, Ca, Fe, K, Mg, Na, Si, Zn (Ba for TCLP) If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.
LCS	One per prep batch. Recovery must meet DoD QSM limits. If the LCS recovery is above the project acceptance limits and there are no detections in the samples, Eurofins TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.

Attachment 11
DoD QSM 5.0 OR 5.1 QC Criteria for Analysis by ICP
(continued)

QSM 5.0 OR 5.1 Table 8. Inorganic Analysis by ICP	
Requirement	**DoD QSM 5.0 OR 5.1 and DOE QSAS 3.0**
Matrix Spike (MS)	One MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. If acceptance criteria are not met, apply J-flag. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
Dilution Test	One per prep batch if MS or MSD fails. Only applicable for samples with concentrations >50x LOQ (prior to dilution). For samples with lower concentrations perform PDS. Five-fold dilution must agree within ± 10% of the original result. Apply J-flag if acceptance criteria not met and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	Perform Recovery Test when dilution test fails or analyte concentration in all samples is <50 x LOQ. Recovery must be within 80-120 % of expected result. If test fails, then run samples by MSA or apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 80 - 120%.
Method of Standard Additions	When dilution or post digestion spike fails <u>and</u> if required by the project. Document use of MSA in case narrative.

Eurofins TestAmerica Denver

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Title: INCREMENTAL SAMPLING METHODOLOGY FOR SOILS AND SEDIMENTS [ASTM D 6323]

Approvals (Signature/Date):

7/6/20	7/6/20
Heather Fiedler Technical Specialist Date	Reed Pottruff Health & Safety Coordinator Date
7/6/20	7/6/20
Roxanne Sullivan Quality Assurance Manager Date	for Scott Hall Laboratory Director Date

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1.0 Scope and Application

1.1 The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of the analytes of interest in the entire parent sample. This is based on the guidance in ASTM Standard D-6323 "Laboratory Subsampling of Media Related to Waste Management Activities," a DoD Quality Systems Manual (QSM) requirement.

1.2 This procedure applies to soils, sediments and other particulate matter. This method is highly dependent on client provided Data Quality Objectives. This procedure presents the laboratory's standard approach, but details at all stages of this procedure can vary from project to project. All project-specific variations must be documented and approved in writing. It is important that the analyst always check special project instructions in the TestAmerica LIMS (TALS) before proceeding.

1.3 TestAmerica has used incremental sampling methodology (ISM) for non-volatile organics (e.g., explosives residues by Method 8330), prior to acid digestion for metals analysis (e.g., Method 3050), and prior to analysis for volatile organics collected in multiple increments in the field and preserved in methanol (e.g., Method 5035). It can be used for a wide range of other analytical methods as well. However, this procedure is not applicable to soil samples to be analyzed for volatile organic compounds in which the entire sample provided by the lab's client is used for a single analysis (see the lab's volatile organics SOP for details).

1.4 This SOP addresses the pre-preparation of samples. The details of the twelve QC Elements, not otherwise addressed, are described in the associated preparation and/or analytical SOPs.

2.0 Summary of Method

2.1 For non-volatile analytes, the entire sample received from the client is air dried to a constant weight. Large non-representative pieces (rocks and twigs that will not pass through the sieve) may be removed manually. Other extraneous materials are removed by sieving. A mortar and pestle or sieve shaker or mechanical disaggregator may be used to break up soil agglomerates during the sieving process. Depending on the analytical method to be used after subsampling, and project objectives, the sample may be ground. The grinding option available at the laboratory is the ring-and-puck mill. A subsample is then taken using a multi-incremental approach.

2.2 ISM for Metals Analysis - the routine approach is to air dry, sieve to sub-10 mesh (2.1 mm), and collect 10 gram subsamples using 30 increments. The expectation is that the variability due to subsampling error will then be no more than 15% relative standard deviation (RSD) (see ASTM D-6323 for explanation and guidance for other acceptable variations). The Method 3050B digestion reagents are then increased proportionally to maintain the same chemistry as is used for 1 gram subsamples.

2.3 ISM for Explosives Analysis - the routine approach is to air dry, sieve to sub-10 mesh, grind (if logged for grinding), and collect 10 gram subsamples using 30 increments. If the samples are from firing points, then ring-and-puck grinding is required. The goal is to

achieve 10% or less RSD from subsampling variability. Further details for explosives are given in SOP DV-OP-0018 and are not discussed in this SOP.

2.4 ISM for Volatile Analysis – the multi-incremental sampling is done in the field, where the 30 increments of 5 grams each are added to a septum-cap bottle containing 200 mL methanol provided by the laboratory. From that point on, the lab's procedure for medium level soils (SOP DV-MS-0002) is followed.

2.5 The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Attachment 1 to this SOP. The Attachment defines the trade off between subsample size, particle size, and the desired level of precision.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

3.2 Sample or Client Sample – refers to the entire quantity of material delivered to the laboratory for testing.

3.3 Subsample – refers to the portion of sample taken in the laboratory for a given analysis. The objective of this procedure is to ensure that the subsample is a reasonably accurate representation of the entire sample.

4.0 Interferences

4.1 If multi-incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory effort entailed in this SOP may add little or no improvement in results.

4.2 Potential loss of lighter semi-volatile compounds (e.g., naphthalene) through the drying and grinding process has not been well studied. Before employing the procedure for such compounds, the possible loss of lighter compounds should be discussed with the client and if possible, investigated before the procedure is performed.

4.3 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or contamination causing misinterpretation of results. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running blanks.

4.4 Contamination by carryover can occur when a low concentration sample is processed immediately following a high concentration sample. For this reason, special care must be taken to follow the equipment cleaning steps.

4.5 As described in this SOP, the lab does not routinely grind samples for metals testing. It is expected that detection limits and reporting limits for some metals would have to be elevated based on long-term blank results if grinding is required.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, TestAmerica Denver Addendum to the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See DV-HS-0010 for details. Personnel operating grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical disaggregator described in Section 6.1.7 during operation, the decibel levels are above 80 decibels, therefore anyone operating the disaggregator must be enrolled in the Hearing Conservation Program and wear hearing protection. While the disaggregator is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.3.2 Operations involving the handling of samples outside of sealed containers, e.g., sieving, are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use in the grinding room, but are optional.

5.3.3 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as “CAT 1”, “CAT 2”, “CAT 3” or “CAT 4” and requires grinding through the ring and puck, contact the Radiation Safety Officer (RSO) immediately.

5.4 Primary Chemical and Material Hazards – cleaning solvents

MATERIAL	HAZARDS	EXPOSURE LIMIT (1)	SIGNS AND SYMPTOMS OF EXPOSURE
Acetonitrile	Flammable Poison	40 ppm – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Acetone	Flammable	1,000 ppm – TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014.

6.1.2 Sieve Shaker – used to facilitate the sieving of large sample volumes.

6.1.3 Ring and Puck - The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe.

6.1.4 Trays – "baker's rack" type stack for air drying soils

6.1.5 Drying tower – custom built tower similar to "baker's rack" type stack for air drying soils, including air drying fans and air filters

6.1.6 Sieves - 10 and 30 mesh, brass for general use, stainless steel for metals testing. Sieves are cleaned after each use by washing with soap and water with a green plastic brillo pad (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kimwipe. Sieves are allowed to dry in a hood prior to use.

6.1.7 Mortar and pestle – Porcelain, various sizes cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. The mortars and pestles are rinsed with 10% acetonitrile in acetone, wiped with a Kimwipe, and allowed to dry in a hood prior to use.

6.1.8 Mechanical Disaggregator – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The stainless steel disaggregator reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical disaggregator is used to break up soil agglomerates but it is not an alternative to the Ring and Puck. The mechanical disaggregator is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 10% acetonitrile in acetone. The Hopper is then wiped dry with a laboratory tissue. The hammers and body of the disaggregator are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Expendable Supplies

6.2.1 Subsampling tools:

6.2.1.1 Scored paper scoops (TAL-0150 and TAL-0150 LARGE from Commodity Management Services)

6.2.1.2 Plastic sample scoops – square-ended

6.2.2 Aluminum foil and aluminum dishes

6.2.3 Parchment paper to line trays for metals testing

6.2.4 Alconox detergent

6.2.5 Ottawa Sand – blank media for organics

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 10% Acetonitrile in Acetone – mix 100 mL of acetonitrile with 900 mL of acetone. This solution is used for rinsing purposes only so exact measurements are not required.

8.0 Sample Collection, Preservation, Shipment and Storage

Container Type	Preservative	Holding Time
Plastic or glass	By individual test *	By individual test *

After air drying, samples can be stored at room temperature.

* - Reference the analytical SOPs.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense

(DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 and 5.1 unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Grinding Blanks

9.2.1 Ring and Puck Grinding Blanks.

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1. Then approximately 200 g of Ottawa sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix “blank”. After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10 g aliquot is then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank with positive results for the same contaminant must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative

9.3 Precision

9.3.1 On a project basis, the lab will discuss precision objectives with the client prior to initiating work. If evaluation of the RSD is needed, the laboratory will need to analyze at least one set of triplicate samples in every preparation batch. In other cases, the lab will employ duplicate matrix spikes and control limits will be

expressed as relative percent difference (RPD).

9.3.2 If the client supplies multiple field samples to use for replicate testing, then the laboratory will compare results to acceptance limits and qualify data if the precision limits are not met. If the replicates are prepared from the single field sample that is dried, ground, and sieved, then the acceptability of each grinding batch can be controlled based on the precision objectives established for the project.

9.4 Other QC samples (method blank, LCS, and MS/MSD) are created after subsampling, and vary depending on the analytical method. See DV-OP-0018 for special QC requirements for explosives, which usually require grinding a standard reference material.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Dry the Samples

10.3.1 The entire contents of the sample container must be processed. It is not acceptable to remove any aliquots until after the sample has been dried, sieved, and ISM performed. If the client requests aliquots to be taken before the sample is dried, sieved, and ISM performed, an NCM should be written to document this was done per client request.

10.3.2 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.3 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.4 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.5 Place the samples in a drying tower, hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.6 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Attachment 3). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing vessel for the analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e., Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.7 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.4.

10.4 Sieve the Samples

10.4.1 Clean the sieves prior to use following the instructions in Section 6.

10.4.2 Some samples may require the use of a mortar and pestle or a mechanical disaggregator to break up dried clumps. Refer to Section 6.1 on how to clean and rinse the mortar and pestles and the mechanical disaggregator before use.

10.4.3 Record the weight of the entire dried sample in the Worksheet tab in TALS. This is a requirement for DoD QSM 5.0 and 5.1.

10.4.4 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples to prevent cross-contamination.

10.4.5 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.6 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle or, as an alternative, use the mechanical disaggregator. Be sure to break up all soil so that it can pass through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.7 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.8 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve. Then a lid or another receiver pan for a second sample is placed on top. The stack is then

clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation were removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.9 If metals or any other analyses are requested on the sample prior to grinding, perform ISM on the portion of the sample that passed through the sieve at this time before proceeding to any grinding steps in Section 10.6. Refer to Method Comments, Sample comments, and Login Comments for instructions if any other tests besides metals analyses are to be performed on an un-ground aliquot before proceeding to Section 10.6

10.5 Incremental Sampling Methodology for Metals and other methods requested on un-ground material.

10.5.1 Select an appropriate subsample container. For metals analyses, a 100 mL digestion cup is appropriate. For organic methods, an amber glass container is appropriate. Reach out to the departments performing the analysis for guidance on selecting the appropriate subsample container.

10.5.2 Remove the cap from the appropriate subsample container and place on a balance and tare. The entire sieved sample is spread out on a sheet of parchment paper to a 1 cm thickness.

10.5.3 Using a subsampling tool (described in section 6.2.1), take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of sieved material.

10.5.3.1 For methods 6010B, 6010C, 6020, and 6020A a 10 g - 11 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 10 g - 11 g aliquot per sample in case re-digestion is needed.

10.5.3.2 For methods 7471A and 7471B, a 3 g - 3.3 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 3g - 3.3g aliquot per sample in case re-digestion is needed.

10.5.3.3 For other methods, refer to method comments, sample comments, and login comments, or to the departments performing the analyses for instructions regarding aliquot size.

NOTE: Sub-out ISM samples will need to aliquoted into amber 40mL VOA vials and delivered to sample receiving with the appropriate paperwork. Aliquot size will vary and depends on the analysis needed.

10.5.4 Record the sample weight on the ISM Worksheet described in Attachment 2.

10.6 Grinding

The instructions in this section are to be used as a general procedure when grinding is requested prior to extraction and analysis for any method. Reference DV-OP-0018 for details on grinding samples for explosives analysis.

10.6.1 Ring and Puck Mill Grinding

10.6.1.1 See Section 6.1 on how to clean the ring and puck dish.

10.6.1.2 If the sample is logged for ring and puck grinding, a grinding blank per Section 9.3.1 consisting of baked Ottawa sand will be processed through the ring and puck dish before each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

10.6.1.3 After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

10.6.1.4 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after the whole sample has been ground. The entire sample **must** be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample for five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by pinching some between two fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

10.6.1.5 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to not perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has flour like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. A NCM should be written to document the deviation from the source method and the reasoning.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the

bath is shallow enough so that water does not get inside the dish.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined without mixing, it has been shown through Duplicate/Triplicate QC results that the sample is non-homogenous. To re-homogenize the sample, lay the sample out on foil/parchment paper and use provided scoop to mix and spread the sample into a 2-D slabcake.

10.7 Incremental Sampling after grinding.

10.7.1 Remove the cap from a 40 mL amber vial or other appropriate subsample container (refer to Section 10.5.1 for guidance) and place on a balance and tare. The entire ground sample is spread out on a sheet of parchment paper or aluminum foil to a 1 cm thickness.

10.7.2 Using a subsampling tool (described in Section 6.2.1), take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material.

10.7.2.1 For explosives a 10 g to 11 g aliquot is required for each sample and each MS/MSD sample.

10.7.2.2 For other extractable methods a 30 - 33 g aliquot is common, but reference project instructions and method SOPs for more detail.

10.7.2.3 For other methods, refer to method comments, sample comments, and login comments, or to the departments performing the analyses for instructions regarding aliquot size.

10.7.3 Record the sample aliquot weight on the ISM Worksheet.

10.8 Maintenance

10.8.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.8.2 When excessive wear is noted, replace the hammers in the Mechanical Grinder.

10.8.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.8.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.9 Troubleshooting

Low recoveries for Tetryl in the explosives grinding LCS may be indicative of high temperatures during grinding. Review the cooling step noted in Section 10.6.1.5 in order to minimize the effect of the heat generated during the grinding process.

11.0 Calculations

Relative Standard Deviation

$$RSD = \frac{S}{\overline{X}}$$

Where: S = standard deviation

$\overline{X}$ = mean

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1 - 2 the laboratory reporting Limit (RL) and carried through the entire preparation and analytical procedure. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

The use of organic solvents to complete the equipment cleaning steps is minimized. Quantities are limited to residues on equipment that quickly evaporate in a hood.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual, *Waste Management and Pollution Prevention*.

14.2 The following waste streams are produced when this method is carried out:

- Solid Waste – Waste Stream S
- Flammable Solvent Waste – Waste Stream C

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 "Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples," USEPA, November 2003.

15.2 "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

15.3 DoD Quality Systems Manual Version 5.0, 2009. Appendix B, Table 3.

15.4 DoD Quality Systems Manual Version 5.1, 2017. Table B-23.

16.0 Method Modifications

Item	Method	Modification
N/A	N/A	*No method modifications specified.*

17.0 Attachments

Attachment 1: Consideration of Fundamental Error in Selecting MIS Options
Attachment 2: ISM Worksheet
Attachment 3: ISM Constant Weight Worksheet

18.0 Revision History

Revision 13, dated 6 July 2020

- Removed sections that prompted analyst to place samples in bags to re-homogenize after puck milling.

Revision 12, dated 3 March 2020

- Annual Reveiw
- Updated copyright information

Revision 11, dated 11 December 2018

- Minor formatting and language corrections throughout
- Replaced all references to “mechanical grinder” to read “mechanical disaggregator” to distinguish more clearly between grinding/milling and soil disaggregation.
- Added new part numbers for preferred subsampling tool to section 6.2.1.
- Added DoD 5.1 reference information to section 9.1.2.
- Added new drying tower to equipment section 6 and to section 10.3.5.
- Removed note in section 10.3.7 which allowed for skipping constant weight analysis for non-DOD samples.
- Added section 10.4.3 to meet DoD 5.1 requirement that dried sample is weighed prior to sieving.
- Removed notes regarding DoD 5.1 specific requirements from section 10.4.6 and 10.4.8, and incorporated these specific requirements into standard procedure for all samples. These requirements include weighing and retaining material that does not pass through the sieve.
- Added wording to 10.4.9 to allow for non-metals tests that are not ground.
- Added sections 10.5.1 and 10.7.1 regarding selection of appropriate subsample container.
- Changed “100 mL digestion cup” in section 10.5.2 to “the appropriate subsample container.”

- Corrected wording in 10.5.3 to read “the layer of sieved material” rather than “the layer of ground material.”
- Added sections 10.5.3.3 and 10.7.2.3 regarding aliquot sizes for methods other than metals.
- Changed language in section 10.7.2 from “a square ended spatula” to “a subsampling tool described in section 6.2.1.”
- Removed section 10.9.1 comment regarding securing ring and puck as it no longer requires securing. Added note about troubleshooting low tetryl recoveries.
- Revised MDL policy reference in section 12.1.

Revision 10, dated 3 October 2017

- Updated section 2.1 to clarify air drying to a constant weight.
- Updated section 8.0 to reflect the secondary storage option for air dried samples.
- Updated section 10.3.6 and 10.3.7 to demonstrate drying to a constant weight.
- Updated section 10.5.2 to reflect ISM Sub-out practices.
- Added attachment 3.

Revision 9, dated 28 February 2017

- Removed all references of the Ball Mill from the body of the instructions.
- Updated Section 3.1 to reference DV-QA-003P and QAM for general terms
- Updated Section 9.1 to be consistent with other SOPs
- Added current Sections 10.1 and 10.2 – NCM reference and instructions
- Updated Section 12 to include current MDL, LOQV, DOC and Training information
- Updated solid waste stream from D to S in Section 14.2

Revision 8, dated 29 February 2016

- Added aluminum dishes to Section 6.2
- Added Section 7.1 and 7.2
- Added what corrective action is used for Ring and Puck Grinding blanks that have hits in Section 9.4.1, as this differs from the corrective action for Ball Mill.
- Changed the acceptable weight range in Section 10.3.2.2 from 3-3.05 g to 3-3.3 g to be consistent with the 10% provided for other methods.
- Added more description to Section 10.4.2.2 to instruct the analyst not to wash the Ring and Puck dish at this step.
- Removed Section 10.6.5 that provided instruction about maintenance of the centrifuge as this equipment is not used in this SOP.
- Removed all revision histories 2010 and earlier (available upon request)

Revision 7, dated 28 February 2015

- Annual Technical Review
- Reformatted the SOP
- Added detail to Section 5.3.1 about the Humbolt mechanical grinder.
- Added information to Section 5 about the hazards of grinding radioactive samples.

- Revised Section 9.4.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
- Revised Section 10.6 to include maintenance on the centrifuge.
- Added Attachment 2: ISM Worksheet

Revision 6, dated 05 February 2014

- Annual Technical Review
- Edited Section 6.1, subsection “Ball Mill” to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans.
- Edited Section 6.1, subsection “Sieves” to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
- Added a comment to Section 9 stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
- Removed Acceptance Criteria information to Section 9. This information can be found in the analytical SOPs.
- Added a NOTE in Sections 10.4.1.1 and 10.4.2.4 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding containers and then later re-combined.
- Added Section 10.6 Maintenance and Section 10.7 Troubleshooting per DoD QSM 5.0.

Revision 5, dated 31 January 2013

- Annual Technical Review
- Added Section 1.4 to address the 12 QC Elements.
- Updated Section 2.3 and 10.4.1 to allow the use of Ball Mill grinding. The laboratory successfully completed a Method Validation for Ball Mill grinding and therefore is able to offer this to all clients.
- Section 6 was updated to include cleaning procedures for sieves, Ring and Puck dishes, Ball Mill stones, Mortar & Pestles, and Paint Cans.
- Section 2.1, Section 6, and Section 10.2.2 were updated to include the mechanical grinder that can be used in place of mortar and pestle for samples that do not require metal testing.
- Section 9.3.3 was updated to include acceptance criteria for Grinding Blanks for DoD samples.
- Section 10.3.1 was updated to instruct the analysts to aliquot the samples directly into 100mL digestion cups for metals analysis.
- Removed Attachment 2: How to Batch Samples in LIMS.

Revision 4.2, dated 31 January 2012

- Removed all references to Multi-Incremental Subsampling which is now trade-marked.
- Updated Section 6.1 to reflect the correct number of small and large grinding stones used in the Ball Mill grinding of samples.
- Updated Attachment 2.

Revision 4.1, dated 20 January 2011

- Added detail about the electronic temperature recording device that records the temperature of the room.
- Revised procedure to state that during the one-minute cooling time, the dish will be placed in a shallow ice water bath to facilitate cooling.
- Revised Attachment 2 to include the method Dry_Grind and more details on how to batch samples that are logged for both MULTI_INC and grinding methods.

Earlier revision histories have been archived and are available upon request.

Attachment 1

Consideration of Fundamental Error in Selecting MIS Options

The following formula given in ASTM D-6323 was used to produce the table that follows.

$$S^2 = 18 * f * e * d^3 / M_s$$

where,

S^2 = the relative variance of the contaminant concentration due to the fundamental error

f = shape factor, a dimensionless number, a value of 0.5 can be taken as typical (Pierre Gy, 1982)

e = the population's average density (g/cm^3). For this table a typical soil density of 2.5 g/cm^3 was used.

d = the diameter of the largest particle in centimeters, and

M_s = the mass of the sample in grams

Sample Mass and Maximum Particle Size to Achieve a Desired RSD

Subsample Mass (g)	Sieve Size (US Standard Mesh)	At 5% RSD Max Size (cm)	At 10% RSD Max Size (cm)	At 15% RSD Max Size (cm)
0.1	35	0.02	0.04	0.05
1	18	0.05	0.08	0.10
2	13	0.06	0.10	0.13
5	12	0.08	0.13	0.17
10	10	0.10	0.16	0.22
30	7	0.15	0.24	0.31
50	6	0.18	0.28	0.37
100	5	0.22	0.35	0.46

Attachment 2

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	Sample	Method --->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
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Attachment 3

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial Date/time 1	Analyst Initial Date/time 2	Dry?		Analyst Initial Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
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Title: Microwave Extraction of Solid Samples by Method [SW-846 3546]

Approvals (Signature/Date):

5/4/20	5/3/20
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5/4/20	5/4/20
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1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing the sample and organic solvent. This procedure achieves analyte recoveries equivalent to those from soxhlet or sonications methods, but uses less solvent. This SOP is based on SW-846 Method 3546.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate solvents and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 15 g, is solvent extracted using a microwave extractor.

3.0 Definitions

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as

a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Sodium sulfate is not used in the extraction vessel. This is because salts are known to super heat when exposed to microwave energy. Samples are extracted without the addition of sodium sulfate, but the extracts are dried with sodium sulfate after the extraction, before concentration of the extracts. If the sample is excessively wet the aliquot can be divided among two or three extraction vessels and the extracts combined prior to concentration.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 Paint chips are an especially difficult matrix to extract. Oftentimes the paint chips dissolve or partially dissolve in solvents and therefore can ruin glassware and extraction vessels. It is the laboratory's experience that paint chips are best extracted by method SW-846 3580 instead of 3550C or 3546.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 A post-run cool down must be used after each extraction to prevent the possibility of operator burns. Pressure builds up in the closed vessel at high temperatures. Care should be taken when opening the vessel when it is above room temperature.

5.1.2 Samples that contain metal fragments or metal components of any kind should not be extracted by this procedure. These samples should be extracted by method SW-846 3550C instead. Care should be taken to inspect samples carefully as they are aliquotted.

5.1.3 Eye protection that satisfies ANSI Z87.1 (as described in the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [(1)]	Hazards	Exposure Limit [(2)]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous. It is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Equipment

6.1.1 Microwave extractor. Mars5: MarsExpress™CEM MARS® and Microwave extractor. Mars6: MarsExpress Plus™ CEM MARS®

At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

6.1.2 Microwave extraction vessels. 75 mL Teflon™ Express vessels with stopper and cap (CEM Corp.) in addition to 110 mL borosilicate glass tubes accompanying 110 mL Teflon™ Express Plus vessels with stopper and cap (CEM Corp).

6.1.3 Hand wrench to tighten the caps on the extraction vessels.

6.1.4 MARS 40 position carousel (CEM Corp) and 20 position carousel (CEM Corp)

6.1.5 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.

6.2 Supplies

6.2.1 Media bottles, 100 mL or 250 mL capped with aluminum foil.

6.2.2 Stainless steel conical funnels

6.2.3 Ashless cellulose filter paper

6.2.4 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.

6.2.5 Metal spatulas or tongue depressors.

6.2.6 Solvent dispenser pump.

6.2.7 Filter flask.

6.2.8 Vacuum pump.

6.2.9 Washing tool for Teflon™ extractor vessels. This tool is a long thin sponge-like brush.

6.2.10 40 mL VOA vials and caps

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Methylene chloride – Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.2 Acetone - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Hexane - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1

before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Baked Sodium Sulfate, 12-60 mesh - Heat sodium sulfate in a 400°C oven for at least four hours. QA personnel post the list of approved lots at solvent storage areas.

7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.

7.6 35% Nitric Acid – Dilute concentrated (70%) Nitric Acid 1:1 in water.

7.7 Standards - Please reference SOP DV-OP-0020 and WI-DV-0009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A[2]	Glass with Teflon-lined lids	15 grams	Cool, ≤ 6°C	None	SW-846
Wipes for Method 8082A[2]	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	15 grams	Cool, ≤ 6°C	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	14 days	SW-846

[1] Exclusive of analysis.

[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require the 14 day holding time for Method 8082. The states of Alabama, California, Colorado, Connecticut, Nevada, New Jersey, Pennsylvania, and Rhode Island require the 14 day holding time for Method 8082.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

9.4.1 A method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

9.4.2 The method blank consists of 15 g of baked Ottawa sand free of any of the analyte(s) of interest.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

9.5.1 At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.5.2 The LCS consists of 15 g of baked Ottawa sand to which the analyte(s) of interest are added at known concentration.

9.5.3 Method AK102 requires LCS and a LCSD for every batch for every spike compound.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.6.2 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

9.6.3 DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must also be documented as a nonconformance, with a cause and corrective action described.

10.3 Critical Procedural Considerations

10.3.1 As stated throughout this SOP, analysts must review the LIMS Method

Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.3.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample.

NOTE: Rotate glassware; do **not** use specific glassware, equipment or positions for the MB and LCS/LCSD.

10.4 Periodic cleaning.

10.4.1 **Mars5 Express Microwave Extractor. CEM Mars** At least once every four weeks, the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the User Directory with the settings that follow:

- Sample Type: Inorganic
- Control Type: Ramp to Temperature
- Power: 100%
- Ramp: 5 minutes to 180°C
- Hold: 10 minutes

10.4.2 **Mars6 Express Plus Microwave Extractor. CEM Mars** At least once every four weeks, the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the Classic Method, "Cleanup2" with the settings that follow:

- Sample Type: Inorganic
- Control Type: Ramp to Temperature
- Stage 1
- Power: 1600%
- Ramp: 15 minutes to 180°C
- Hold: 10 minutes
- Temperature guard : 200°C

10.4.3 Fill each tube with 30 mL of the nitric acid solution described in Section 7 and cap tightly. Place the tubes in the carousel, then run the "Clean Method"

10.4.4 Allow the vessels to cool, and then dispose of the nitric acid in waste stream J. Rinse the vessel with DI water three times.

10.4.5 Fill each tube with 30 mL of 1:1 Methylene Chloride: Acetone solution and

cap tightly. Place the tubes in the carousel, then run the "Clean Method" again.

10.4.6 Allow the vessels to cool, and then dispose of the solvent in waste stream CA. Allow the vessels to air dry.

10.5 Assemble and Clean the Extraction Tubes Immediately Before Use.

10.5.1 If the microwave tube, cap, or plugs are wet, pre-rinse with acetone.

10.5.2 Rinse the microwave tube, cap and plug twice with methylene chloride. The plugs can be placed in a large glass jar to help facilitate the rinse.

10.5.3 Discard the solvent in the correct waste stream.

10.6 Aliquot Samples

10.6.1 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. If the sample is a wipe, transfer the wipe to the extraction vessel.

10.6.2 Label microwave vessel with the sample ID, method, and batch number. The label needs to be flat.

NOTE: For method 8270 borosilicate glass tubes are to be used in housing the sample in addition to required QC; respectively. The Glass tubes will be inserted into the retaining vessel and capped for extraction.

This method is performed using the Mars6 Microwave only

10.6.3 Do not use specific vessels or carousel positions for the MB and LCS.

10.6.4 For each MB and LCS sample, weigh 15 g to 17 g of baked Ottawa sand into labeled VOA vials or similarly clean glass intermediate containers with a lid. Record a nominal weight of 15 g in the initial volume field, but record the actual weight to the nearest 0.1 g in the notes column.

10.6.5 For each sample and MS/MSD, weigh 15 g to 17 g of sample into labeled VOA vials or similarly clean glass intermediary containers with a lid. Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

NOTE: For wipe samples, the original sample containers that the wipes are received in should be used in place of the intermediary sample containers described above.

10.6.6 Cap the intermediary sample containers either with the appropriate lid or aluminum foil.

10.6.7 Place the labeled intermediary sample containers on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Checklists (See WI-DV-0009).

10.7 Prepare a bottle with a bottle-top dispenser with the appropriate solvent(s).

10.7.1 Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B
- SW-846 8015C
- SW-846 8015D
- Low-Level NDMA (8270D_SIM_LL)

10.7.2 For soil extraction by all other methods, the solvents used are acetone and methylene chloride. These are added separately.

10.7.3 For wipe samples by method 8081 and 8082, the solvent used is hexane.

10.7.4 For wipe samples by method 8270 SIM, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.8 Add Surrogate and Spike Solutions

NOTE: The standards should be allowed to come to room temperature before spiking the samples.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009.

10.8.1 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples. Document the standards and pipette(s) used on the benchsheet.

10.8.2 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard (see WI-DV-0009) to the intermediary container for each field sample and QC sample. Verify the ID of the standard used on the benchsheet.

10.8.3 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard (see DV-OP-0009) to the intermediary container for each field sample and QC sample. Verify the ID of the standard used on the benchsheet.

10.9 Making sure not to overflow the intermediary sample container, remove the cap, and slowly add approximately 15 mL of the appropriate solvent to the container. See below for the appropriate solvent:

NOTE: The solvent should be added as soon as possible after the addition of the surrogate and spiking standards to prevent loss of the more volatile compounds.

10.9.1 15 mL of methylene chloride is added to the container for soil and wipe

samples for the following methods:

- SW-846 8015B, 8015C, and 8015D
- Low-Level NDMA (8270D_SIM_LL)

10.9.2 15 mL of acetone is added to the intermediary container for all other soil samples.

10.9.3 15 mL of hexane is added to the container for wipe samples by method 8081 and 8082, the solvent used is hexane.

10.9.4 15 mL of 1:1 methylene chloride and acetone is added to container for wipe samples by method 8270 SIM.

10.10 Mix the contents of the intermediary sample container using a spatula or wooden tongue depressor for 30 seconds. Then, cap the intermediary sample container and use a vortex mixer to mix the contents of the intermediary sample container for an additional 30 seconds. This mixing must generate a thoroughly wetted and disaggregated sample slurry. If clay clumps or other sample aggregation is evident after 1 minute of combined manual and vortex mixing, sand may be added to facilitate disaggregation, followed by an additional minute of mixing (manual + vortex as described above). If sand is added during this step, document this in an NCM.

10.11 Transfer the mixed/wetted sample to a microwave extraction vessel using three 5 mL methylene chloride rinses. This will bring the total combined solvent volume to 30 mL.

NOTE: The solvent should completely cover and saturate the sample. Additional solvent may be needed depending on the matrix of the individual sample. The sample and solvent must not fill more than 2/3 of the vessel.

NOTE: If the sample matrix appears to be unusual, or especially wet, the combined sample/solvent mixture can be equally divided between two or three separate microwave extraction vessels. The vessels will be extracted independently, but the extracts will be re-combined before concentration. This will prevent the extraction vessels from over-heating and venting if the sample is unusually wet, oily, or bulky (if a 15 g aliquot would fill the tube more than ¾ full). If the sample is split into two or three separate vessels, document this in an NCM.

NOTE: For method 8270, the mixed/wetted sample or QC will be transferred into borosilicate glass tubes. The glass tubes are then inserted into the retaining vessel and capped for extraction.

10.12 Seal the vessels by placing the plug on top of the vessel, small side down, and hand tighten the cap over the plug.

NOTE: Care should be taken to ensure that the plug, the cap, and the threads of the vessel are clean of any material or debris.

10.13 After being sealed, the vessels must be inverted several times to ensure that the

material is well mixed and saturated. It is recommended that when extracting with 100% methylene chloride to vent and re-cap the vessels before continuing to relieve excess pressure and thereby preventing the vessels from venting during the extraction.

NOTE: 8270 Samples extracted using the borosilicate glass tubes will not be inverted, as this will result in the sample + solvent spilling out of the glass tube into the retaining vessel.

10.14 Load vessels into the carousel.

10.14.1 There must be at least 8 vessels in the carousel. Adding blank vessels with sand and solvent may be necessary.

10.14.2 Balance the tubes around the carousel to ensure that all samples are exposed to an equal amount of energy during the extraction. See Attachment 1 for details. Only samples using the same extraction solvent should be placed in the same carrousel and run at the same time.

10.14.3 For the vessels to be correctly loaded in the carousel the cap should completely touch the top of the carousel with no other part of the extraction vessel visible.

10.15 Place the carousel into the microwave, making sure that it sits on the turning apparatus correctly. The carousel should be able to rotate. Close the door.

10.16 Mars5 Express: The Method Menu screen should indicate “Start Current Method” as being 3546 Full Xpress. Press the green “Start/Pause” button to begin the extraction.

NOTE: If a different method is shown, go to the “Load Method” on the menu screen. Choose “User directory” and place the cursor on the desired method. Press the “Home” button to return to the main menu, where the test highlighted will appear under the “Start Current Method”.

10.16.1 The method is under the User Directory with the settings that follow:

- Sample Type: Organic
- Control Type: Ramp to Temperature
- Power: 100% (1600 W)
- Ramp: 20 minutes to 115°C
- Hold: 10 minutes

10.17 Mars6 ExpressPlus: The “One Touch Method” menu should be selected. Next, the “CEM 3546 glass 110C” method should be selected. At the bottom right hand corner of the screen should be a green “start” selection. Press the green “Start” button to begin the extraction.

NOTE: If a different method is shown, press the back arrow, found on the bottom left hand of the screen” until you reach the appropriate menu.

10.17.1 The method is under the CEM 3546 glass 110C" program with the settings that follow:

- Sample Type: Organic
- Control Type: Ramp to Temperature
- Stage 1
- Power: 500-1500W
- Ramp: 15 minutes to 110°C
- Hold: 15 minutes

10.17.2 When the extraction is complete, the vessels will need to return to room temperature prior to opening the vessels. The microwave will indicate the approximate temperature of the vessels.

CAUTION: If the carousel is removed from the microwave before the vessels are at room temperature, do NOT open the vessels. The vessels may be placed in a rack outside of the microwave to cool down.

10.17.3 The microwave contains a solvent sensor that will indicate the presence of solvent in the microwave and will stop the extraction. To minimize this, care needs to be taken not to overfill the vessel and to properly cap and tighten the vessel prior to extraction. If the solvent sensor indicates the presence of solvent, open the door and inspect the tops of the tubes for evidence of a solvent leak. If solvent has vented or leaked out of an extraction vessel, the sample must be re-aliquotted and the extraction started over. It is best to re-aliquot the sample into two or three separate extraction vessels to prevent over-heating again. Document this in an NCM.

10.18 Assemble and Clean Filter Funnels and Media Jars.

10.18.1 Without gloves on, fold a 18 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 100 mL or 250 mL media bottle.

NOTE: For low-level NDMA samples by method 8270D_SIM_LL, use designated glass funnels instead of the stainless steel funnels and instead of re-usable media jars, use disposable amber bottles. This is done to prevent contamination.

10.18.2 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with the extraction solvent (see Section 10.7), so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the

rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.18.3 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.18.4 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.18.5 Discard the solvent in the correct waste stream.

10.19 Filter the Extracts

10.19.1 After the extraction method is complete and the vessels reach room temperature, quantitatively transfer the entire sample through solvent rinsed sodium sulfate funnels and into the media jar. The quantitative transfer is performed by rinsing the microwave extraction vessel at least three times with solvent.

NOTE: The quantitative rinse is vital in order to achieve good recoveries. The rinses should be significant enough that when done, the extract volume is between 75 mL and 100 mL.

NOTE: If the sample aliquot was split between two or three tubes, the extracts from all the tubes shall be combined at this time. Filter all of the extracts through the same sodium sulfate funnel and collect in the same media jar.

NOTE: During the 8270 extraction, it has been noted that solvent may be found in the retaining vessel after extractions. This contains analytes of interest and should be filtered into the funnel with the rest of the sample. Quantitatively rinse the glass tube; DO NOT rinse the retaining vessel.

10.19.2 Once the solvent has completely drained into the collection apparatus, rinse the funnel contents with 10 to 20 mL of additional solvent. Dispose of the solid sample and sodium sulfate into Waste Stream D and cap the media jar with aluminum foil.

NOTE: For 8270 extractions, dispose of the glass tube

10.20 If the extract contains visible solids, it will be necessary to filter the extract again prior to concentration.

10.21 Store the extract refrigerated at $\leq$ 6°C until concentration. Ensure that the extracts in 1:1 Methylene chloride:acetone are placed in a flammable rated refrigerator.

10.22 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

10.23 All glassware and microwave tubes, plugs, and caps are washed according to DV-OP-0004.

10.24 Maintenance

10.24.1 As needed, wipe out the inside and outside of the microwave with a damp cloth.

10.24.2 See Section 10.4 for vessel cleaning.

10.24.3 At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

10.25 Troubleshooting

10.25.1 If it appears that the solvent sensor is malfunctioning, ensure that the sensor is aligned at a 45 degree upward angle on the back of the unit.

10.25.2 The snorkel vent should be set inside of a hood, but care should be taken so that the opening is not blocked. Make sure the snorkel does not press against the back of the hood.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health

and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 1:1 MeCl2:Acetone – Waste Stream CA

15.2.3 Flammable solvent – Waste Stream C

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Nitric Acid Waste – Waste Stream J

15.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3456 Microwave Extraction, Revision 0, February 2007.

16.2 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water", Manchester Environmental Laboratory, Dept of Ecology, State of Washington.

17.0 Method Modifications:

17.1 SW-846 Method 3546 calls for samples to be either air-dried and ground or mixed with sodium sulfate prior to extraction. This procedure does not call of the air-drying of samples unless requested by the client as this may lead to loss of the more volatile compounds. Sodium sulfate is not used in the extraction vessel, rather the extracts are dried with sodium sulfate after extraction and prior to concentration. Salts are known to superheat when exposed to microwave energy.

17.2 SW-846 Method 3546 calls for samples to be aliquoted on a balance capable to weighing to 0.01 g. This SOP calls for a balance capable to weighing to 0.1 g as this is sufficient to report data to 3 significant figures.

17.3 SW-846 Method 3546 Section 1.4 states "2-20 g of material is usually necessary and can be accommodated by this extraction procedure." This SOP calls for 30-33 g of material.

17.4 SW-846 Method 3546 Section 11.7 states "Add approximately 25 mL of the appropriate solvent system to the vessel." This SOP calls for the addition of 25-30 mL of solvent.

17.5 Method NWTPH-Dx calls for samples to be extracted by method SW-846 3550C. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method NWTPH-Dx is a possible determinative method by this procedure.

18.0 Attachments

Table 1: Determinative Methods Using Microwave Extraction

Attachment 1: Proper Carousel Loading

19.0 Revision History

- Revision 10, May 4, 2020
  - Annual Review.
- Revision 9, April 25, 2019
  - Removed all references to Alaska methods AK 102 and AK 103 as these will now be conducted with Sonication in DV-OP-0016 exclusively.
  - Annual Technical Review
- Revision 8, March 6, 2018
  - Changed nominal sample weight from 30 grams to 15 grams in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA. This change is reflected in sections 2.0, 8.0, 9.4, 9.5, and 10.6.
  - Changed all references to WI-DV-009 to correct document ID: WI-DV-0009.
  - Modified sections 10.6-10.11 to specify the use of the intermediary sample container required in order to implement the microwave extraction best practices and standardization procedure provided by corporate QA.
  - Modified section 10.7.2 to clarify that acetone and methylene chloride are added separately in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA.
  - Modified, added, and/or rearranged sections 10.9-10.11 in accordance with the microwave extraction best practices and standardization procedure provided by corporate QA. This change involves modifying the addition of solvents, mixing of the sample/solvent mixture, and transferring from an intermediary container into the microwave extraction vessel.
  - Updated sections 6.1.1, 6.1.2, 6.1.4, 10.4.1, 10.4.2, 10.6.2, 10.6.5, 10.11, 10.14, 10.15, 10.15.1, 10.17.1, 10.17.2 with notes to reflect the usage of the new microwave for 8270 FS, HSL list analytes only.
- Revision 7, January 31, 2017
  - Annual Technical Review
  - Added paragraph to Section 3.0 referencing the QAM for general definitions
  - Added paragraph to Section 6.0 to record IDs of pipettes and equipment
  - Updated language in Section 9.6.3 requiring LCSDs when no MS/MSD
  - Added note to Section 10.3.2 on rotating glassware/equipment/positions
  - Added current Section 13.2 defining LOQV
- Revision 6, January 31, 2016

- Annual Technical Review
- Updated Section 9.1 to contain verbiage consistent with other SOPs
- Added Section 9.6.3 regarding DoD MS/MSD requirements
- Changed the “Clean Method” frequency from two to four weeks in Section 10.4.1
- Changed the waste stream from C to CA in Section 10.4.5
- Section 10.5.2 changed the rinse requirement to be performed twice.
- Added Section 10.6.3 instructing not to use specific vessels or positions for the MB and LCS.
- Modified Section 10.6.4 weight recording requirements
- Added Section 10.6.6 – cap with aluminum foil
- Added the documentation of the standards and pipette used in Section 10.8.1
- Clarified the need to punch a hole in foil when spiking to Section 10.8.2 & 10.8.3
- Clarified the process for adding solvent to vessels in Section 10.9
- Added the requirement to place 1:1 Methylene chloride:acetone extracts in a flammable rated refrigerator to Section 10.18
- Revised Section 13.1 – Method Detection Limit Study (MDL)
- Revised Section 13.2 – Demonstration of Capabilities
- Revised Section 13.3 - Training Requirements
- Updated Section 17.4 to reflect the addition of 25-30 mL of solvent
- Archived all revision histories 2010 and earlier

- Revision 5, January 31, 2015
  - Annual Technical Review
  - Reformatted SOP
  - Revised Section 7.4 to remove the requirement to test the sodium sulfate before use. This was done to reflect current practice in CA-Q-S-001-DV-1.
  - Added “NWTPH DRO” to the procedure
  - Revised Section 10.5.2 to state that the plugs and caps can be rinsed in a large glass jar.
  - Added a note in Section 10.15.1 to state that for method 8270D_SIM_LL, designated glass funnels and disposable amber bottles will be used to filter the extracts.
  - Added Sections 16.2-16.5 to list AK102, AK103, and NWTPH methods as references.
  - Removed Section 17.8, redundant with 17.5.
  - Updated Table 1 to reference the correct methods and SOPs.

- Revision 4, January 31, 2014
  - Annual Technical Review
  - Revised Section 1.2 to state that the procedure may be used for additional methods when appropriate solvents are used instead of pH as there are no pH adjustments made in the procedure.
  - Removed TeflonTM lined caps from the Equipment and Supplies list in Section 6 as the lab now uses aluminum foil.
  - Added footnote to the table in Section 10 stating some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require 14 day hold time for method 8082A.
  - Revised Section 9.1.2 to state that this procedure meets all criteria of DoD QSM 5.0.
  - Revised Section 9.4 to clarify that one method blank is processed with each batch.

- Removed "Acceptance Criteria" and "Corrective Action" information from Sections 9.4, 9.5, 9.6, and 9.7. This information can be found in the analytical SOPs.
- Added a bullet point in Section 10 to clarify that any deviations discovered after the procedure is performed are to be documented in an NCM.
- Revised Section 10 to remove the instruction to place the label towards the bottom of the vessel. This is not necessary. Also removed the requirement that the label must include the date. The label includes the batch number, which is unique and the date of extraction is recorded in the batch.
- Revised the procedure to state the periodic acid cleaning of the tubes should be done at least once every two weeks instead of weekly.
- Removed methods "NWTPH DRO" and "Okla_DRO" from the procedure. The lab does not perform microwave extraction for these methods at this time.
- Added sub-sections for Maintenance and Troubleshooting to Section 10 per DoD QSM 5.0.
- Added low-level NDMA and 8015D as a possible analytical method to Section 10 and to Table 1
- Removed 8310 as a possible analytical method in Table 1.
- Added Attachment 1 to give instructions on how to properly load the vessels in the carousel.

- Revision 3, January 31, 2013
  - Annual Technical Review
  - Sections 4.2 and 10.5.4 were revised to remove the optional addition of sodium sulfate to the samples before extraction. It was determined that the better option when dealing with wet samples is to split the sample into two or three tubes and re-combine the extracts before concentration.
  - Section 4 was revised to add instructions on how to deal with paint chip samples.
  - Section 5 was revised to add comments about the dangers of metal fragments in samples.
  - Section 6 was revised to include the requirement that the Power Measurement Calibration procedure be performed on the unit every year.
  - Section 8 was revised to update the hold times for Method SW-846 8082A.
  - Section 10.8 was revised to give more detail on how full the extraction vessel should be once solvent has been added.
  - Section 10.13.1 was revised to allow the carousel to be removed from the microwave unit before the vessels are cool so long as the vessels are not opened.
  - Section 10.15.1 was revised to add a note about the importance of quantitative transfers and rinses while filtering the extracts.
  - Section 10.15.1 was revised to add instructions to combine all extracts from samples that were originally split across two or three tubes.
  - Section 15 was revised to include the waste stream CA.
  - Added the Note to Table 1

- Revision 2.0, January 31, 2012
  - Annual Technical Review
  - Updated Section 4.2 and Section 10.5.4 to describe when sodium sulfate should be used in the extraction vessel.
  - Updated Section 6.0 to allow the use of aluminum foil to cap 100mL and 250mL media jars.

- Updated Section 6.1 to include details on computer software and hardware.
- Updated Section 7.0 to include details on the purity of reagents and standards.
- Updated Section 9.1.4 and Section 10.1 to more accurately reflect the NCM process.
- Corrected grammatical and formatting errors
- Updated Section 10.3 to include a solvent cleaning after the weekly acid cleaning.
- Updated Section 10.5.4, Section 10.7.2, and Section 10.7.3 to include an option to split the sample aliquot into two separate microwave vessels.
- Updated Section 10.10 and 10.13.2 to give details on how to prevent vessels from over-heating and venting and steps to be taken if venting does occur.
- Updated Section 10.16 to accurately reflect how the laboratory handles extracts with suspended sediment.
- Updated Section 10.19 to reference SOP DV-OP-0004 on how to clean the microwave vessels.

- Revision 1 dated 01 Jan 2011
  - Added 8270C SIM as a valid determinative method by microwave extraction.
  - Changed the procedure to call for the extract to be filtered thru a conical steel funnel lined with cellulose filter paper instead of a glass funnel with glass wool. This was done to help remove sediment from the extracts.
  - Removed details about the surrogate and spike standards used in the extraction. This information can now be found in DV-OP-0020.
  - Added instructions to Section 7 on how to prepare the nitric acid solution used in the weekly cleaning of the tubes.
  - Changed the solvent used in the extraction of samples for method 8081 and 8082. The samples are now extracted in a 1:1 Mixture of MeCl2:Acetone instead of a 1:1 Mixture of MeCl2:Hexane.
  - Revised the procedure in Section 10.5 for aliquotting samples to state that 30 to 33g of sample should be used instead of 30±2g and that the weight should be recorded to the nearest 0.1g instead of the nearest mg.

Earlier revision histories have been archived and are available upon request.

TABLE 1.

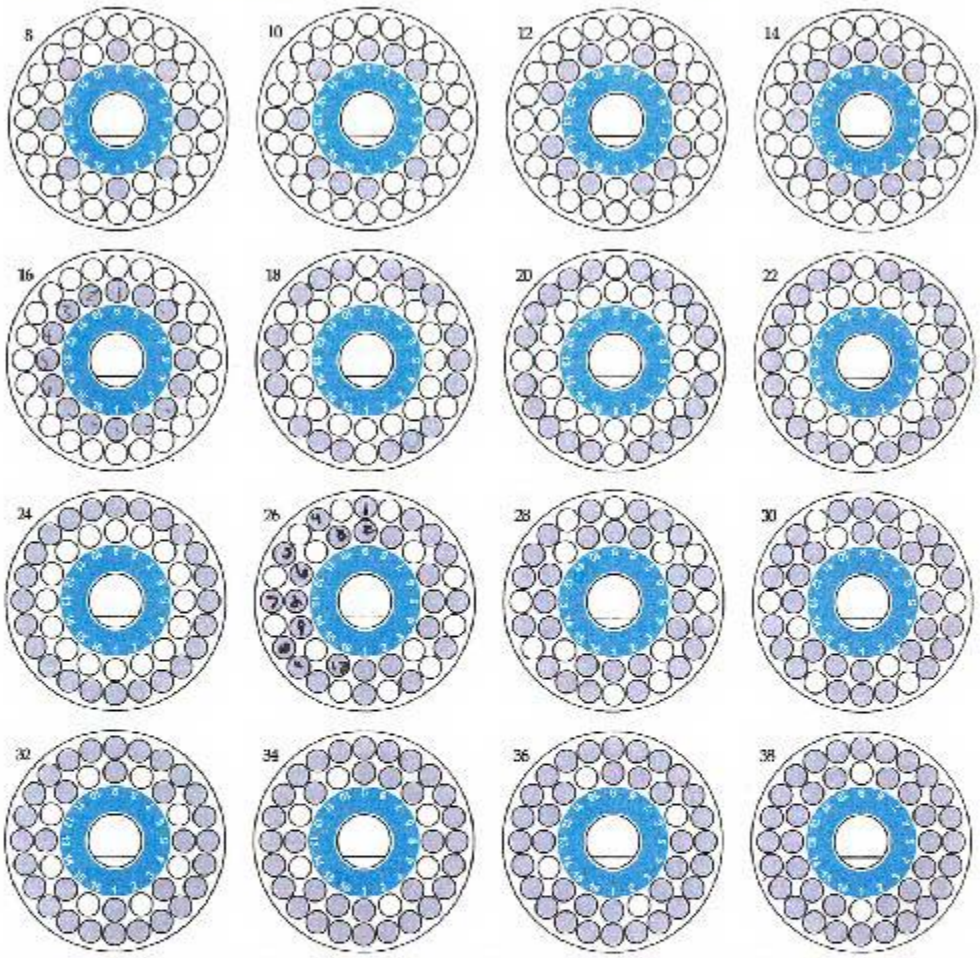
Determinative Methods Using Microwave Extraction

Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D NWTPH-Dx AK102 AK103	DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002
Low-Level NDMA by Isotope Dilution, GC/MS SIM, Large Volume Injection	SW-846 8270C/D SIM	DV-MS-0015

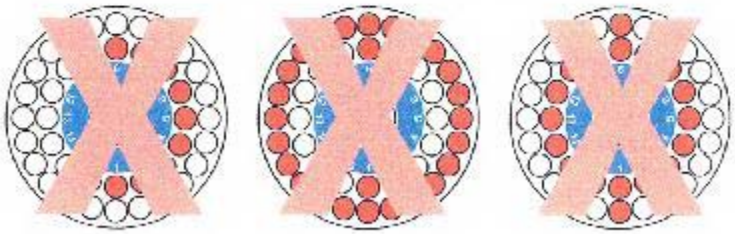
ATTACHMENT 1.

Proper Carousel Loading

Recommended distribution of MARSXpress vessels when turntable is less than capacity



Incorrect distribution: What not to do



eurofins	Always check on-line for validity.	Level:
Document number: T-WC-WI11483	**Colorimetric Sulfide in Waters, Sulfide as H2S, Dissolved Sulfide in Waters by 4500-S2 D-2011, 4500-S2 H-2011, or EPA 376.2**	**Work Instruction**
Old Reference: 1-P-QM-WI-9011642		
Version: 18		Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date 12-MAR-2020	Document users: **6_EUUSLA_ Instrumental Water Quality_Alpkem Analys, 6_EUUSLA_ Instrumental Water Quality_Alpkem Verifi, 6_EUUSLA_Water Quality_Misc Analysis, 6_EUUSLA_Water Quality_Misc Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

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Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
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Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
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Apparatus and Equipment
Reagents and Standards
Reagents for Standardization of Sulfide Standard Intermediate Solution
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 18		**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		**Removed revision logs up to the previous version**
Throughout Document	Updated Instrumentation		Added Gallery Discrete Analyzer Methodology
Reference Modifications	New section		Addresses wavelength
Definitions 3.	Addition		Added ICV
Sample Collection, Preservation and Handling	Clarification		Added amount of zinc acetate to be added for preservation.
Apparatus and Equipment	Clarification		Added balance precision and burette graduations.
Reagents and Standards 2.	Clarification		Added concentration of solution Added Note.
Reagents and Standards 3.	Clarification		Added concentration of solution
Calibration	No longer required		Curves no longer have to be analyst-specific for North Carolina
Calibration	method requirement		Must analyze an ICV
Procedure B.1.e -f.	Additions		CCBs required. LOQ check required for North Carolina samples.
Calculations B	Correction		Added step 2.d.
Quality Control/Quality	Method requirement		Added ICV, ICB, CCB

Assurance	Clarification	Added matrix spike instructions
Quality Control/Quality Assurance	Addition	Added LOQ check requirement for North Carolina samples
Attachments	Updated Instrumentation	Added Gallery Discrete Analyzer Settings

Revision: 17	**Effective Date:**	March 15, 2018
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 P QM-QMA-9017356	Removed revision logs up to the previous version
Title	Correction 40 CFR 136	Removed 4500-S2 F and 4500-S2 B/C Updated to current version (2011)
Reference	Not active method references 40 CFR 136	Removed SM18-SM22 references and SM4500-S2 C Updated to current version (2011)
Reference 3-4.	Clarification	These references are only for specific steps in the process.
Cross Reference	Higher level documents are not required to be referenced	Removed Cross References: 1-P-QM-QMA-9015389,1-P-QM-QMA-9017309, 1-P-QM-QMA-9017313,1-P-QM-QMA-9017328
Reference Modifications	Reference Modifications section not needed.	Removed Reference Modifications Section
Reagents and Standards	Correction Enhancement	Increased amount of sodium sulfide crystals to 3.75g Added instruction for dealing with contamination on crystals. Added warning to avoid introducing excess oxygen.
Reagents and Standards	Enhancement	Moved QC prep from reagents and Standards to Procedure section
Reagents and Standards 3.	Correction - current procedure	Reagent is currently purchased.
Calibration D.3.f.	Correction - method requirement	changed 16030 to 16000 in standardization calculation
Procedure A.2.b	Clarification	added step for mixing contents
Procedure B.1	Enhancement Addition	Moved QC prep from Reagents and Standards to Procedure section Added MS/MSD preparation instructions
Procedure B.8.	Addition	Added instructions for diluting over-range samples
Calculation A.	Addition	added calculation for mg/L sulfide
Calculation B.	Correction	Updated H2S calculation to reflect current method reference.

Reference

1. *Standard Methods for the Examination of Water and Wastewater*, Method 4500 S2 D-2011.

2. *Standard Methods for the Examination of Water and Wastewater*, Method 4500-S2 H-2011.

3. *Standard Methods for the Examination of Water and Wastewater*, Method 4500-S2 B-2011. (flocculation for dissolved sulfide analysis)

4. *Standard Methods for the Examination of Water and Wastewater*, Method 4500-S2 F-2011. (iodometric method for standardization of sulfide)

5. Method 376.2, *Methods for the Chemical Analysis of Water and Wastes*, USEPA 600.

6. Method 376.1, *Methods for the Chemical Analysis of Water and Wastes*, USEPA, 600.

7. *Standard Methods for the Examination of Water and Wastewater*, Method 4500 S2 D-2000.

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-WC-WI10352	Spectronic Genesys 2 and Genesys 10 Vis Spectrophotometers
T-WC-WI10348	Fixed Volume Hand-Held Pipettes
T-WC-WI9897	Adjustable Volume Handheld Pipettes
T-WC-WI10360	Quality Control Data for Wet Chemistry

Scope

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The colorimetric sulfide method is applicable to the measurement of total and dissolved sulfides in drinking, surface, and saline waters and domestic and industrial wastes. The procedure is applicable at sulfide concentrations between 0.1 and 20 mg/L.

Analysis #10293 is used to determine the amount of sulfide as H2S in fresh or salt waters.

Basic Principles

Sulfide reacts with dimethyl *p* phenylene diamine in the presence of ferric chloride to produce methylene blue. Diammonium hydrogen phosphate is added after color development to remove the ferric chloride color. The methylene blue dye is read either on a manual spectrophotometer at a wavelength of 625 nm or on an automated analyzer. The concentration of sulfide is calculated by comparison to a standard curve.

Reference Modifications

SM 4500-S2 D-2011 recommends a 664 nm wavelength. The analyses in this SOP using the 625 nm wavelength meet the method QC requirements. The 625 nm wavelength is recommended by EPA Method 376.2.

Definitions

1. Calibration Blank – reagent water to which all reagents, **except the color indicator reagent**, are added in the same proportions as samples.

2. Method Blank (May also be called a reagent blank) – reagent water that is carried through the same analytical procedure as samples, and to which **all reagents** are added in the same proportions as samples.

3. Initial Calibration Verification standard (ICV) - A second-source standard within the calibration range that is used to determine the accuracy of the calibration. This standard must be analyzed immediately after a calibration curve and must be accompanied by an initial calibration blank (ICB).

Interferences

Oxygen inadvertently added to the sample may volatilize the sulfide so samples for sulfide analysis must be collected with a minimum of aeration.
Interfering substances such as sulfite, thiosulfate, iodide, and other soluble substances are removed with the addition of zinc acetate to form the precipitate, zinc sulfide.

Sample color and turbidity also may interfere. Correct this by running sample blanks.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

NOTE: Adding acid to samples that may contain high concentrations of sulfide must be done in a well-ventilated area to protect laboratory staff from accidental exposure to hydrogen sulfide gas.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples for total sulfide must be collected in a glass container with minimum aeration.
Preserve with 0.2 ml of 2N zinc acetate per 100 ml of sample. Preserve with sodium hydroxide (NaOH) to a pH

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of at least 9. Fill bottle completely and stopper. Refrigerate at 0° to 6°C, not frozen, and analyze within 7 days of collection.

Apparatus and Equipment

1. Spectrophotometer for use at 625 nm with 1 cm cell or equivalent. Refer to *T-WC-WI10352*.
2. Test tubes with screw type lids or equivalent

3. Automatic pipettes. Refer to *T-WC-WI9897*.

4. Analytical balance capable of weighing to 0.01 g.

5. Class A burette with burette stand (50 ml capacity with 0.1 ml graduations)

6. For analysis 14856 The Thermo Fisher Gallery Discrete Analyzer is used.

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same.

1. 1+1 sulfuric acid

   a. In a glass beaker, slowly and carefully add 250 mL of concentrated sulfuric acid (H_2SO_4) to slowly stirring 250 mL of reagent water.

   b. Solution becomes very hot. Allow time to cool.

   c. Store in a glass bottle at room temperature and re-evaluate yearly.

2. Amine-Sulfuric Acid Stock Solution, for Sulfide Determination (Methylene Blue method) (27% w/v N,N-dimethyl-*p*-phenylene diamine oxalate)

NOTE: Currently this reagent is not used. The Amine-Sulfuric Acid working reagent is purchased instead, making the stock solution unnecessary.

If needed, stock solution may be purchased and stored per manufacturer's instructions, or prepared as follows:

   a. Add 50 mL of concentrated H_2SO_4 slowly and carefully to 20 mL of reagent water.

   b. After solution has completely cooled, dissolve 27.0 ± 0.5 g N,N dimethyl *p* phenylene diamine oxalate in this solution.

   c. Cool and dilute to 100 mL with reagent water in a volumetric flask.

   d. Store in amber glass bottle at room temperature.

   e. Stable 1 year. (When this stock solution is diluted and used in the procedure, the reagent blank will first be pink and then turn colorless within 3 minutes. If this doesn't happen, discard reagent and remake with fresh oxalate.)

3. Amine-sulfuric acid working reagent (0.68% w/v N,N dimethyl *p* phenylene diamine oxalate)

   a. Purchased, see manufacturer's label for expiration date.

   b. Or to prepare:

      (1.) Dilute 2.5 mL amine sulfuric acid stock solution to 100 mL with cooled 1+1 H_2SO_4 in a volumetric flask.

      (2.) Store in an amber glass bottle at room temperature.

      (3.) Stable 6 months.

4. Ferric chloride solution

   a. Dissolve 100.0 ± 0.5 g $FeCl_3 \cdot 6H_2O$ in approximately 40 mL of reagent water.

   b. Store in an amber glass bottle at room temperature.

   c. Stable 6 months.

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5. Diammonium hydrogen phosphate solution

a. Dissolve 250.0 ± 0.5 g (NH4)2HPO4 in approximately 500 mL of reagent water.

b. Store in glass bottle at room temperature.

c. Stable 6 months.

6. Sulfide standard stock solution

a. Dissolve 3.75 ± 0.01 g Na2S·9H2O in reagent water and dilute to 500 mL in a volumetric flask.

b. Crystals may have excess water on the surface or a layer of contamination from oxidation products. They may be rinsed and blotted dry before weighing.

c. Concentration of solution is approximately 1000 mg/L.

d. Avoid excess agitation to limit introducing oxygen to the standard

e. Store in glass at 0° to 6°C, not frozen

f. Stable 7 days.

NOTE: A separate source of Na2S·9H2O must be used when preparing the sulfide standard stock solution used for the calibration curve.

7. 2N zinc acetate solution – Purchased. Store at room temperature. See manufacturer's label for expiration date. Used to prepare intermediate sulfide solution.

8. 5N sodium hydroxide solution (NaOH)

a. Dissolve 200 ± 1 g of NaOH in approximately 800 mL of reagent water.

b. Stir well with stir bar. (Solution will get very warm.)

c. Dilute to 1000 mL in a volumetric flask.

d. Store at room temperature and re evaluate yearly. Used to prepare sulfide standard intermediate solution.

9. Sulfide standard **intermediate** solution

a. Pipette 20.0 mL of sulfide stock solution into approximately 800 mL of reagent water in a 1000 mL volumetric flask.

b. Preserve with NaOH to a pH >9 and add approximately 3 mL of purchased 2N zinc acetate solution.

c. Dilute with reagent water to 1000 mL.

d. Prepare fresh and standardize daily. (Concentration of solution is approximately 20 mg/L). Used as spiking solution.

10. 5-mg/L Sulfide working standard:

Where:

X = mL of standardized intermediate sulfide solution (diluted to **200 mL** with reagent water in a volumetric flask) needed to obtain a sulfide working standard solution equal to 5 mg/L concentration.

$$X = \frac{1000}{\text{mg/L actual concentration of intermediate sulfide solution}}$$

NOTE: 5 mg/L sulfide working standard must be prepared fresh daily.

11. 6N aluminum chloride solution – Purchased, or equivalent. Store at room temperature. See manufacturer's label for expiration date. Used for dissolved sulfide sample preparation.

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12. Intermediate Cal Standard for analysis 14856 (10 mg/L)

a. Add 1 mL of the 1000mg/L Cal Std to a 100mL volumetric flask and bring to volume with reagent water.

b. Prepare fresh daily

13. CCV/LCS for analysis 14856 (1.0 mg/L)

a. Add 0.1 mL of the 1000 mg/L QC Std to a 100 mL volumetric flask and bring to volume with reagent water.

b. Prepare fresh daily.

Reagents for Standardization of Sulfide Standard Intermediate Solution

1. Hydrochloric acid (1+1)

a. Mix equal portions of concentrated hydrochloric acid (HCI) and reagent water.

b. Store at room temperature and re evaluate yearly.

2. Starch indicator – Purchased. Store at room temperature. See manufacturer's label for expiration date.

3. Potassium iodide (KI) crystals – Purchased. Store at room temperature and re evaluate yearly.

4. 0.025N potassium bi-iodate

a. Dry KH(IO3)2 for at least 2 hours at 104° ± 1°C and cool approximately one hour in a desiccator.

b. Dissolve 0.8124 ± 0.0002 g KH(IO3)2 in reagent water and dilute to 1000 mL in a volumetric flask.

c. Store in amber glass bottle at room temperature.

d. Stable 1 year.

5. 0.1N sodium thiosulfate – Purchased. Store at room temperature. See manufacturer's label for expiration date.

6. 0.025N sodium thiosulfate (Na2S2O3)

a. Dilute 250 mL of 0.1N Na2S2O3 (purchased) to 1000 mL with reagent water.

b. Hold time is determined by manufacturer's expiration date for 0.1N Na2S2O3.

c. Store at room temperature.

d. Standardize weekly against 0.025N potassium bi iodate.

7. 0.025N iodine solution

a. Dissolve 20 to 25 g KI in approximately 800 mL of reagent water and add 3.20 ± 0.05 g iodine.

b. Allow plenty of time to dissolve and then dilute to 1000 mL with reagent water in a volumetric flask.

c. Stable 1 year.

d. Store in amber glass bottle at room temperature.

e. Standardize weekly against 0.025N sodium thiosulfate.

Calibration

A. Balances must be calibrated each day before use.

B. Pipettes must be checked for accuracy daily.

C. Spectrophotometer must be calibrated as per *T-WC-WI10352*.

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D. Standardization:

Record all standardization in the solution standardization logbook.

1. 0.025N Sodium thiosulfate

a. Dissolve approximately 2.0 g KI in an Erlenmeyer flask with approximately 150 mL reagent water. Add a few drops of concentrated H2SO4. Swirl.

b. Add by pipette 20 mL of 0.025N potassium bi iodate.

c. Dilute to approximately 200 mL with reagent water. Immediately titrate the liberated iodine with 0.025N Na2S2O3 titrant to a pale yellow color.

d. Add a few drops of starch solution and titrate to the first colorless endpoint.

e. Perform duplicate trials and average the results.

$$Na_2S_2O_3N = \frac{(20mLKH(IO_3)_2) \times (0.025N\,KH(IO_3)_2)}{mL\ of\ sodium\ thiosulfate\ titrated}$$

2. 0.025N Iodine

a. Pipette 10 mL of 0.025N iodine solution into an Erlenmeyer flask.

b. Add approximately 90 mL of reagent water.

c. Titrate immediately with the Na2S2O3 (previously standardized) to a pale yellow color.

d. Add a few drops of starch solution and titrate to the first colorless endpoint.

e. Perform duplicate trials and average the results.

$$IodineN = \frac{(mL\ of\ Na_2S_2O_3\ titrated)\ (standardized\ normality\ of\ Na_2S_2O_3)}{10.0\ mL\ iodine\ solution}$$

3. 20-mg/L intermediate sulfide solution

Add reagents to the Erlenmeyer flask in this order to ensure no sulfide gas is liberated before the titration is performed.

a. Pipette 10 mL of iodine solution (previously standardized) into an Erlenmeyer flask.

b. Add approximately 10 mL of reagent water and approximately 2 mL of 1+1 HCI.

c. Pipette 100 mL of well-mixed, precipitated 20 mg/L intermediate solution into the flask, discharging it under solution surface. Swirl.

d. Titrate immediately with Na2S2O3 (previously standardized) to a pale yellow color.

e. Add a few drops of starch solution and titrate to the first colorless endpoint.

f. Perform duplicate trials and average the results.

Standardization Calculation:

mg/L sulfide = [(A x B) - (C x D)] x 16000 / 100 ml of standard

Where:

A = mL of iodine solution
B = Normality of iodine solution

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C = mL of $Na_2S_2O_3$ solution
D = Normality of $Na_2S_2O_3$ solution

E. Curve (zero spectrophotometer with calibration blank)

1. Prepare a calibration curve every 3 months or when a new reagent is prepared using the sulfide concentrations listed below. Include a reagent blank as part of the curve. Alternate volumes are acceptable as long as the final concentrations remain the same.

Concentration (mg/L)	mL of 5-mg/L standard (diluted to a final volume of 100 mL)
0.00	0 (reagent blank)
0.10	2
0.50	10
1.00	20
1.50	30
2.00	40

2. Zeroing the spectrophotometer with a calibration blank:

a. Prepare a calibration blank by pipetting 7.5 mL of reagent water into a 10 mL test tube.

b. Add by pipette, 0.5 mL of 1+1 H_2SO_4 and 0.15 mL (3 drops) ferric chloride solution. **Mix immediately by inverting slowing only one time**. Wait 3 to 5 minutes for color development.

c. Add by pipette 1.6 mL diammonium hydrogen phosphate solution to test tube. Slowly invert test tube 3 times. Wait 10 minutes for color development.

d. Zero the spectrophotometer with the calibration blank **at 625 nm using a 1 cm cell.**

3. Pipette 7.5 mL of each standard curve point into a 10 mL test tube.

4. To test tubes, add by pipette, 0.5 mL of amine-sulfuric acid working reagent and 0.15 mL (3 drops) ferric chloride solution. **Mix immediately by inverting slowing only one time**. Mixing too vigorously causes low results by releasing hydrogen sulfide gas before it has had time to react. Presence of sulfide will be indicated by the development of blue color. Wait 3 to 5 minutes for complete color development.

5. Add by pipette 1.6 mL diammonium hydrogen phosphate solution to each test tube. Slowly invert test tube 3 times. Wait 10 minutes.

6. Read absorbance on spectrophotometer **at 625 nm using a 1 cm cell**.

7. Plot calibration curve of mg/L sulfide concentration vs. absorbance using least squares fit. The correlation coefficient of the curve must be ≥ .995.

8. A second-source Initial Calibration Verification Standard (ICV) and an Initial Calibration Blank (ICB) must be analyzed immediately after the calibration curve is completed. See Quality Assurance/Quality Control section of this SOP for more information.

9. For analysis 14856:

a. The Gallery Discrete Analyzer will prepare a set of calibration points using the 10 mg/L Intermediate Cal standard and analyze them at 660 nm. The calibration concentrations the gallery prepares are listed below.

Cal ID:	**mg/L**
SG-5	2.0
SG-10	1.0
SG-20	0.50
SG-25	0.40
SG-100	0.10
SG-0	0.00

b. The calibration for this analysis is performed on the instrument daily.

Procedure

Record data in a raw data logbook following instructions in QA-SOP11913.

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A. Sample Preparation:

1. To remove interferences prior to analysis, zinc acetate preserved samples for total sulfide must be prepared in the following manner:

a. Mark on the bottle the volume of the sample.

b. After the zinc sulfide precipitate settles out and a clear liquid phase is **visible**, siphon down to the precipitated area (being careful not to pull any of the zinc sulfide precipitate out).

c. Refill to the original sample volume mark with reagent water.

d. Re-suspend the precipitate before withdrawing a portion for analysis.

e. Sample is no longer considered preserved and needs to be analyzed as soon as possible.

f. Samples without a visible precipitated layer must be analyzed without this pretreatment step.

2. Prepare samples for **soluble (dissolved) sulfide** in the following manner:

NOTE: If sample is entirely free from visible suspended solids, dissolved sulfide equals total sulfide. This means the sample is analyzed without the following pretreatment step.

a. Fill the bottle with sample (pH adjusted to >9 with NaOH) and add approximately 4 drops(0.2mL) of 6N aluminum chloride solution.

b. Rotate back and forth about a transverse axis vigorously for 1 minute or longer to flocculate contents.

c. Vary the amounts of aluminum chloride and NaOH, if needed, to get good clarification without using excessively large amounts and to adjust the pH to between 6 and 9.

d. Let settle until a reasonably clear supernatant is visible.

e. Analyze supernatant as soon as possible.

B. Sample Analysis

1. QC Preparation Instructions

a. Method blank - analyze 7.5 ml of reagent water.

b. Analyze 7.5 ml of the 1 mg/l sulfide LCS/ICV/CCV standard for the LCS(D).

c. Prepare the MS and MSD by spiking 7.5 ml of sample with 0.2ml of the intermediate sulfide standard.

d. CCV preparation

(1). 0.5 mg/l sulfide CCV
(a). pipette 10 ml of the 5 mg/l sulfide working standard into a 100-ml volumetric flask
(b). Bring up to volume with reagent water
(c). Prepare fresh daily. Use as prepared for CCV

(2). 1 mg/l sulfide LCS/ICV/CCV
(a). Pipette 20 ml of the 5 mg/l sulfide working standard into a 100-ml volumetric flask
(b). Bring up to volume with reagent water
(c). Prepare fresh daily. Use as prepared for LCS/ICV/CCV

(3). 1.5mg/l sulfide CCV
(a). Pipette 30 ml of the 5 mg/l sulfide working standard into a 100-ml volumetric flask
(b). Bring up to volume with reagent water
(c). Prepare fresh daily. Use as prepared for CCV

e. ICB/CCB preparation - analyze 7.5 ml of reagent water

f. For North Carolina samples, analyze an LOQ check standard at the level of the LOQ (0.3 mg/l), each day samples are analyzed.
Pipette 4 ml of the 20 mg/l sulfide standard to 250 ml reagent water.

2. Zeroing the spectrophotometer with a calibration blank:

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a. Prepare a calibration blank by pipetting 7.5 mL of reagent water into a 10 mL test tube.

b. Add by pipette, 0.5 mL of 1+1 H2SO4 and 0.15 mL (3 drops) ferric chloride solution. **Mix immediately by inverting slowing only one time**. Wait 3 to 5 minutes for color development.

c. Add by pipette 1.6 mL diammonium hydrogen phosphate solution to test tube. Slowly invert test tube 3 times. Wait 10 minutes.

d. Zero the spectrophotometer with the calibration blank, **at 625 nm using a 1 cm cell.**

3. Using a wide-tipped pipette, place 7.5 mL of sample or 7.5 mL of a suitable dilution of sample each into two 10 mL test tubes, one for the sample and the other for a sample blank.

4. Sample:

a. Add by pipette, 0.5 mL of amine-sulfuric acid working reagent and 0.15 mL (3 drops) ferric chloride solution.

b. **Mix immediately by inverting slowing only one time**. Mixing too vigorously causes low results by releasing hydrogen sulfide gas before it has had time to react. Presence of sulfide will be indicated by the development of blue color.

c. Wait 3 to 5 minutes for complete color development.

5. Sample blank:

a. Each sample requires a sample blank to subtract interferences.

b. Add by pipette, 0.5 mL of 1+1 H2SO4 and 0.15 mL (3 drops) ferric chloride solution.

c. **Mix immediately by inverting slowing only one time.**

d. Wait 3 to 5 minutes for complete color development.

6. Add by pipette 1.6 mL diammonium hydrogen phosphate solution to test tube (samples and sample blanks). Slowly invert test tube 3 times. Wait 10 minutes.

7. Read absorbance on spectrophotometer **at 625 nm using a 1 cm cell**.

8. If the concentration result of the absorbance is greater than the highest concentration on the curve, then the sample must be diluted and reanalyzed to produce an absorbance contained on the curve.

a. Diluent is reagent water.

9. Automated Discrete Analyzer (Gallery) (analysis 14855)

a. An instrument calibration is analyzed daily for the Gallery Discrete Analyzer.

b. The instrument calibration analyzes five standards of know concentration and a zero concentration point (see table calibration section for concentration of the standards). An Initial Calibration Check (ICV 1.0 mg/L) and an Initial Calibration Blank (ICB) is analyzed with each calibration.

c. When the calibration standards have been analyzed, check the linearity of the calibration. The coefficient must be>0.995 for the curve to be acceptable.

d. If the calibration and calibration check standards pass all criteria continue to analyze samples with the appropriate laboratory control standard (LCS) and preparation blank (PB). Analyze a calibration verification standard (CCV) and calibration blank (W) between every ten injections.

e. Any sample that falls above the highest standard of the full scale calibration will automatically be flagged and diluted by the instrument. The Instrument method is designed to automatically dilute sample up to a concentration of 100 mg/L H2S. Any sample that exceeds this concentration will need to be manually diluted and added to the instrument schedule.

g. End the run with a calibration verification standard and calibration blank

Calculations

A. Colorimetric Sulfide and Dissolved Sulfide

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Subtract the sample blank absorbance from the sample absorbance and obtain concentration of sulfide in mg/L from calibration curve. Multiply this value by sample dilution factor to calculate mg/L sulfide concentration in sample.

mg/L sulfide = A x DF

Where:
A = concentration from the sulfide curve

DF = Dilution factor

B. Sulfide as H_2S

1. pH and specific conductance are needed to determine sulfide as H_2S.

2. The specific conductance is used to determine if a sample is freshwater or saltwater.

   a. The definition of saltwater for this analysis is a specific conductance > 50,000 umhos/cm.

   b. If the specific conductance is less than 50,000 umhos/cm, the sample is considered freshwater.

      (1). Estimate the sulfide as H_2S for freshwater using Figure 4500-S^2:III SM 4500-S^2 H-2011.

   c. If the specific conductance is greater than 50,000 umhos/cm, the sample is considered saltwater.

   d. Multiply the percentage of sulfide as H_2S by the result from the dissolved sulfide analysis.

      (1). Sulfide as H_2S Saltwater Calculation

salinity = $1.6 \times 10^{-5}C$

Where: C = conductivity (umhos/cm) from Table 2330:I SM 4500-S^{2-} H-2011

$\alpha_{H_2S} = [H_2S]/S_T = 1/(10^{pH-pK'sw} + 1)$

Sulfide as H_2S (mg/L) = $\alpha_{H_2S} \times S_T$

Where:

S_T = total dissolved sulfide concentration

pK'_{SW} is determined from Table 4500-S^{2-} :III using temperature and salinity.

Statistical Information/Method Performance

The method detection limit (MDL) is determined annually by following the procedure outlined in *QA-SOP11892*. For this analysis, the MDL study must be analyzed over a minimum of 3 days.
The quality control acceptance windows are generated annually by following the procedure outlined in *QA-SOP11896*.

Quality Assurance/Quality Control

1. Manual colorimetric analysis

Batch size is limited to 20 samples or less.

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A method blank and a 1.0-mg/L laboratory control standard (LCS) must be analyzed on each batch.

The method blank must be less than half the LOQ for this analysis.

An Initial Calibration Verification Standard (ICV; midrange check standard of 1.0 mg/L) must be analyzed immediately following a calibration curve. The ICV must have a recovery between 90% - 110%.

If the ICV standards do not meet the acceptance criteria, then prepare new standards and rerun. If the ICV is still out of specification, then analyze a new calibration curve and repeat the ICV standards. If the acceptance criteria is not met, then see your supervisor for direction.

A continuing calibration verification (CCV) standard and a calibration blank (CCB) must be analyzed at the beginning of each batch, after every ten samples and at the end of each batch. The concentration of the CCV must alternate in a repeating wave pattern: 0.5 mg/L, 1.0 mg/L, 1.5 mg/L, 1.0 mg/L, 0.5 mg/L, etc. On each batch, at least one 0.5 mg/L CCV must be analyzed to validate the curve. The acceptance range for all CCVs is 90% to 110%.

The CCB must be analyzed immediately after every CCV. All samples must be bracketed by an acceptable CCV and CCB. The acceptable range for the blank is not greater than ½ the LOQ. If the blank does not meet this requirement, the cause for the unacceptable result must be determined, if possible. Corrective action must be taken and a CCV/CCB reanalyzed.

A matrix spike (MS), a matrix spike duplicate (MSD), and a sample matrix duplicate must be analyzed with each batch. For the manual colorimetric analysis, prepare the MS and MSD by spiking 7.5 ml of sample with 0.2 ml of the intermediate sulfide standard.

When sample volume does not permit the analysis of either a sample matrix duplicate or an MS and an MSD, then a 1.0 mg/L LCSD must be analyzed to demonstrate method precision.

A second sample matrix duplicate is not required unless a batch contains an NPDES sample(s) from South Carolina and the batch contains more than 10 samples.

For North Carolina samples, each day that samples are analyzed, a LOQ check standard must also be analyzed. The acceptable range is 50 to 150% of the true value.

Samples from West Virginia require a usable precision measurement for each batch. Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: an MS/MSD, a non-program deleted duplicate, or an LCSD.

Refer to *T-WC-WI10360* for requirements that fall under Chapter 4 of Standard Methods for the Examination of Waters and Wastewaters, 22nd Edition.

If any of the QC samples do not meet required specifications, refer to *T-WC-WI10360*. Follow guidelines in *T-WC-WI10360* for outlier QC data and for corrective action. See LIMS for current quality control acceptance windows.

2. Discrete Analysis (analysis 14856)

A calibration curve of five standards and a blank must be run daily. The correlation coefficient of the curve must be >0.995. If this is not met, the calibration is unacceptable and must be rerun.

An initial calibration verification standard (ICV) (CCV 1.0 mg/L H_2S) must be run immediately after every calibration. The acceptable range is ± 10% of the true value.

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A calibration blank (ICB) must be run after the ICV. The acceptable result is < the method detection limit.

A calibration verification standard (CCV 1.0 mg/L H_2S) and a calibration blank (CCB) must be run every 10 injections. The acceptable range for the CCV is ± 10% of the true value. An acceptable calibration blank result is < 1/2 the reporting limit.

An LCS (1.0 mg/L H_2S) must be prepared and analyzed with every batch of samples prepared. For waters, the acceptable range of the LCS is 90% to 110% of the true value.

A batch blank (PB) must be prepared and analyzed with every batch of samples prepared. The acceptable result is < the method detection limit.

Attachment:
SM 4500-S2 2011 Figure 3 (.pdf)
Sulfide.docx (.docx)

T-WC-WI10348 Fixed Volume Hand-Held Pipettes
T-WC-WI10352 Genesys 30 Spectrophotometer
T-WC-WI10360 Quality Control Data for Wet Chemistry
T-WC-WI9897 Adjustable Volume Handheld Pipettes

End of document

Version history

Version	Approval	Revision information
16	02.JUN.2015	
17	01.MAR.2018	
18	12.MAR.2020	

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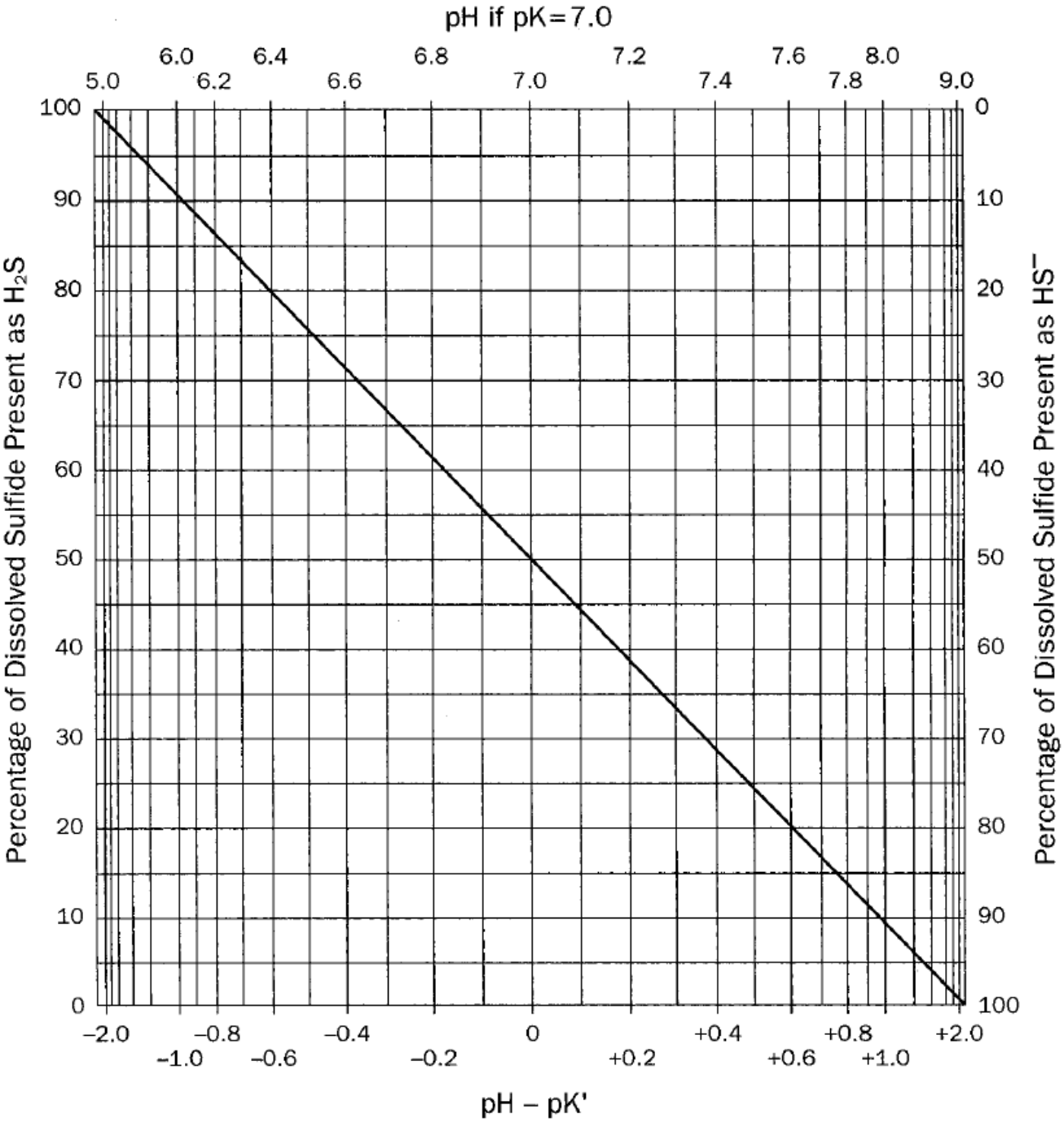

Figure 4500-S^{2-}:3. Proportion of H_2S and HS^- in dissolved sulfide.

Sulfide

Test Parameters

Sample	Reagent Amine	Reagent FeCl	Incubate	Blank Sample	Reagent DHP	Incubate	Absorbance Measurement
120 µL	8 µL	2 µL	240 sec		26 µL	600 sec	620 nm

Total Volume 156 µL

eurofins	Always check on-line for validity.	Level:
Document number: **T-WC-WI10422**	**Acid Volatile Sulfide in Solids**	**Work Instruction**
Old Reference: **1-P-QM-WI-9013392**		
Version: **14**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **06-FEB-2015**	Document users: **6_EUUSLA_Water Quality_Misc Analysis, 6_EUUSLA_Water Quality_Misc Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

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Revision Log

Revision: 14	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 P QM-QMA-9017356	Removed revision logs up to the previous version
Procedure A.11.	Reflects current referenced procedure	Reduced time to 10 minutes

Revision: 13	**Effective Date:** Jul 30, 2014	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 P QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects re-identification of documents in EtQ Reflects current industry standards	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers Changed refrigeration temperature from 4±2°C to 0° to 6°C, not frozen
Reagents and Standards	Requirement	Added instructions for the making of and use of deaerated reagent water
Calibration C.1.	Requirement	Added instructions to use deaerated reagent water when preparing curve standards
Procedure A.	Requirement	Added instructions to use deaerated reagent water during the generation of H_2S

Procedure A.5.	Enhancement	Added step 5 to reinforce the need to minimize sulfide oxidation
Procedure B.1.b	Requirement	Added instructions to use deaerated reagent water for analyzing sulfide
Procedure B.2.	Requirement	Added instructions to use deaerated reagent water for analyzing sulfide

Reference

1. EPA-821-R-91-100 "Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment", December 1991

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1 P QM PRO 9015535	Quality Control Data for Wet Chemistry
1 P QM QMA 9015389	Balance, Syringe, Pipette Verification
1 P QM QMA 9017309	Determining Method Detection Limits and Limits of Quantitation
1 P QM QMA 9017313	Establishing Control Limits
1 P QM QMA 9017328	Reagents and Standards
1 P QM QMA 9021767	Laboratory Notebooks, Logbooks And Documentation for Environmental Testing

Scope

This method is applicable for the determination of acid volatile sulfide (AVS) in sediment samples and water samples.

Background Information

If selected simultaneously extracted metals (SEM) are requested, the remaining acidified sediment sample is filtered and analyzed. The filtering and SEM analysis are done in a separate department, so the procedure is not addressed in this method.

Basic Principles

A measured amount of sample is purged under nitrogen gas and then acidified with hydrochloric acid (HCl). Any AVS in the sample is converted to hydrogen sulfide (H_2S), which is purged from the sample and trapped in an aqueous solution of antioxidant buffer. An ion selective electrode determines the amount of sulfide and the concentration is then calculated by comparison to a standard curve.

Interferences

Any oxygen inadvertently added to the sample may prematurely volatilize the sulfide.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and

pollution prevention.

Converting AVS in the sample to H_2S with acid must be done in a hood or well-ventilated area to protect laboratory staff from accidental exposure to H_2S gas.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.

Analysts are considered proficient when they have successfully completed a Demonstration of Capability. Demonstration of Capability consists of four check standards that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Collect samples for AVS analysis in a glass container with no headspace. Refrigerate at 0° to 6°C, not frozen and analyze within 14 days of collection.

To prevent unwanted aeration, do not homogenize samples before analysis.

Use bottle code #74 when possible.

Apparatus and Equipment

1. Each analytical setup requires the following:

   a. Stirring apparatus

   b. Tri-neck distillation flask with round-bottom and ground-glass joints or equivalent

   c. Addition funnel with pressure equalizing tube and ground glass joints or equivalent

   d. Ground-glass stopper and keck clips or equivalent

   e. Gas washing bottle and scrubber assembly (trap) or equivalent

   (Assembly of this apparatus is shown in Figure 1.)

2. Flowmeter or equivalent

3. Analytical balance, or equivalent. Refer to LOM SOP LAB 208

4. pH/mV meter or ion-selective meter compatible with the ion selective electrodes

5. Sulfide ion selective electrode

6. Double junction reference electrode

NOTE: Follow manufacturer's instructions for use of the meter and electrodes.

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same. Refer to 1 P QM QMA 9017328 for the proper labeling and documentation of reagent preparations.

All water and reagents used in this method must be free of dissolved oxygen and sulfide. Deaerate reagents immediately before use by deaerating with oxygen free nitrogen.

1. Deaerated reagent water - Vigorously bubble reagent water with oxygen free nitrogen for approximately one hour. This will remove dissolved oxygen from the reagent water. Make fresh each day of use.

2. Hydrochloric acid (1 + 1) – Mix equal portions of concentrated HCl and deaerated reagent water. Store at room temperature and re evaluate yearly. Deaerate immediately before use.

3. Sulfide antioxidant buffer (SAOB) – Dissolve 160.00 ± 0.1 g sodium hydroxide (NaOH) pellets in approximately 1400 mL of deaerated reagent water. Solution becomes very warm; cool to room temperature. To NaOH solution, add and dissolve 148.90 ± 0.1 g disodium ethylenediaminetetraacetic acid (EDTA) and 70.46 ± 0.1 g ascorbic acid. Dilute to 2000 mL with deaerated reagent water in a volumetric flask. Store in a glass or plastic container at room temperature. Stable 1 week. Deaerate immediately before use.

4. Starch indicator – Purchased. Store at room temperature. See manufacturer's label for expiration date.

5. Potassium iodide (KI) crystals – Purchased. Store at room temperature and re evaluate yearly.

6. 0.025N potassium bi-iodate – Dry $KH(IO_3)_2$ for at least two hours at 104° ± 2°C and cool approximately one hour in a desiccator. Dissolve 0.8124 ± 0.0002 g $KH(IO_3)_2$ in reagent water and dilute to 1000 mL in a volumetric flask. Store in amber glass bottle at room temperature. Stable 1 year.

7. 0.1N sodium thiosulfate – Purchased. Store at room temperature. See manufacturer's label for expiration date.

8. 0.025N sodium thiosulfate ($Na_2S_2O_3$) – Dilute 250 mL of 0.1N $Na_2S_2O_3$ (purchased) to 1000 mL with reagent water. Hold time is determined by the manufacturer's expiration date for 0.1N $Na_2S_2O_3$. Store in glass or plastic container at room temperature. **Standardize weekly** against 0.025N potassium bi iodate.

9. 0.025N iodine solution – Dissolve 20 to 25 g KI in approximately 800 mL of reagent water and add

3.20 ± 0.05 g iodine. **Allow plenty of time to dissolve** and then dilute to 1000 mL with reagent water in a volumetric flask. Stable 1 year. Store in amber glass bottle at room temperature. **Standardize weekly** against 0.025N sodium thiosulfate.

10. Stock sulfide standard solution (Concentration of solution is approximately 50 µmoles/mL) – Dissolve 6.00 ± 0.01 g $Na_2S{\cdot}9H_2O$ in deaerated reagent water and dilute to **500 mL** in a volumetric flask. Store in amber glass bottle at 0° to 6°C, not frozen. Stable 1 week. **Standardize daily** against 0.025N iodine solution.

a. Blank, LCS, and MS/MSD Preparation

(1) For the blank and LCS, weigh 10 ± 0.5 g sea sand or glass beads.

(2) Before closing the system pipette 10 mL of the 50 µmoles/mL stock standard solution to the LCS, MS and MSD flasks.

NOTE: A separate stock sulfide standard solution must be prepared using a different source of $Na_2S{\cdot}9H_2O$ when preparing a calibration curve.

Calibration

A. Balances must be calibrated each day before use. Refer to 1 P QM QMA 9015389.

B. Standardization:

1. Sodium thiosulfate – Dissolve approximately 2.0 g KI in an Erlenmeyer flask with approximately 150 mL reagent water. Add a few drops of concentrated H_2SO_4 and pipette 20 mL of 0.025N potassium bi iodate. Dilute to approximately 200 mL with reagent water. Immediately titrate the liberated iodine with 0.025N $Na_2S_2O_3$ titrant to a pale yellow color. Then add a few drops of starch solution and titrate to the first colorless endpoint. Perform duplicate trials and average the trials.

$$Na_2S_2O_3\ N = \frac{(20\ mL\ KH\ (IO_3)_2) \times (0.025N\ KH\ (IO_3)_2)}{mL\ of\ sodium\ thiosulfate\ titrated}$$

2. Iodine – Pipette 10 mL of 0.025N iodine solution into an Erlenmeyer flask and add approximately 90 mL of reagent water. Titrate with the $Na_2S_2O_3$ (previously standardized) to a pale yellow color. Then add a few drops of starch solution and titrate to the first colorless endpoint. Perform duplicate trials and average the trials.

$$Iodine\ N = \frac{(mL\ of\ Na_2S_2O_3\ titrated)\ (normality\ of\ Na_2S_2O_3)}{10.0\ mL\ iodine\ solution}$$

3. Stock sulfide standard solution – Pipette 10 mL of iodine solution (previously standardized) into a 125 mL Erlenmeyer flask. Add by volumetric pipette 2 mL of the sulfide standard stock solution and then add 5 mL of 1 + 1 HCl into the flask. Titrate with the $Na_2S_2O_3$ (previously standardized) to a pale yellow color. Then add a few drops of starch solution and titrate to the first colorless endpoint. Perform duplicate trials and average the trials.

$$Sulfide\ (\mu moles/mL) = [(N\ of\ Iodine)(10\ mL\ of\ iodine) - (N\ of\ Na_2S_2O_3)(mL\ of\ Na_2S_2O_3\ titrated)]\ x\ 250$$

C. Calibrate the sulfide ion-selective electrode system by analyzing a calibration curve each day the electrode is used.

Concentrations of curve points (µmoles)
20
250
500
750
1000

1. The exact concentration of the sulfide standard stock solution will determine the mL needed to prepare these sulfide curve points.1. Into a 100 mL volumetric flask, pipette the mL of sulfide standard stock solution needed. Add 50 mL of SAOB reagent and dilute to volume with deaerated reagent water. Mix well. Pour entire contents into a 150 mL glass beaker.

2. Rinse sulfide and reference electrodes into waste container and blot dry with absorbent tissue. Immerse electrodes into the contents of the beaker. Stir gently.

3. Allow electrode response to stabilize and take the relative millivolt (Rel mV) readings.

4. Plot calibration curve of µmoles sulfide concentration vs. Rel mV readings using a log plot in the statistics program in LIMS. The correlation coefficient of the curve must be ≥ 0.995.

NOTE: When analyzing samples from the state of North Carolina, each analyst performing the analytical procedure must produce a standard curve (Ref 15A NCAC 2H .0805 (a) (7) (l)).

Procedure

A. Generation of H_2S Procedure:

Record data in a raw data logbook following instructions in 1 P QM QMA 9021767.

1. Prepare and use deaerated reagent water by removing dissolved oxygen by vigorously bubbling with oxygen free nitrogen for approximately one hour.

2. Deaerate reagents immediately before use by deaerating with oxygen free nitrogen.

3. Place 50 mL of SAOB reagent and 30 mL of deaerated reagent water into the gas washing bottle.

4. Weigh 10 ± 0.5 g (or a suitable portion) of sample directly into the tri neck distillation flask and record the amount.

5. Perform steps 6 -10 immediately after weighing each sample to minimize exposure to air.

6. Add 100 mL deaerated reagent water and a magnetic stirring bar to the flask.

7. In a hood, assemble the glassware apparatus as shown in Figure 1.

8. Place 20 mL of deaerated 1 + 1 HCl into the addition funnel with the stopcock closed.

9. Check all the clips and securely close the system.

10. Begin purging the system with nitrogen gas.

11. Adjust the flowrate to 40 mL/min and continue the flow of nitrogen gas for at least 10 minutes.

12. Begin magnetically stirring the sample and open the addition funnel stopcock to add 20 mL of 1 + 1 HCl directly into the flask.

13. Adjust the flowrate to 20 mL/min and continue the flow of nitrogen gas for 1 hour. Continue stirring throughout this part of the test.

14. After the hour is finished, turn off the nitrogen gas flow and disconnect the scrubber assembly. Analyze the contents according to the following procedure.

B. Analysis of Sulfide Procedure:

1. Transfer the contents of each gas washing bottle into a 100 mL volumetric flask.

   a. Ensure all liquid is included by forcing the liquid through the glass frit with pressure from a pipette bulb.

   b. Ensure quantitative transfer by rinsing the trap with deaerated reagent water.

2. Bring the solution to volume with deaerated reagent water. Mix well.

3. Pour entire contents into a 150 mL glass beaker.

4. Rinse sulfide and reference electrodes into waste container and blot dry with absorbent tissue.

5. Immerse electrodes into the contents of the beaker. Stir gently.

6. Allow electrode response to stabilize and record the relative millivolt (Rel mV) readings.

7. Calculate the AVS concentration from the standard curve by using the LIMS statistics program.

Calculations

$$\mu moles/g \quad acid\ volatile\ sulfide = \frac{concentration\ from\ curve\ (\mu moles)}{sample\ weight\ (g)}$$

Statistical Information/Method Performance
The method detection limit (MDL) is determined annually by following the procedure outlined in 1 P QM QMA 9017309.

The quality control acceptance windows are generated annually by following the procedure outlined in 1 P QM QMA 9017313.

Quality Assurance/Quality Control
A method blank and a 50 µmoles/g laboratory control standard (LCS) must be analyzed on each batch. Batch size is limited to 20 samples or less.

A continuing calibration verification (CCV) standard must be analyzed at the beginning of each batch, after every ten samples and at the end of each batch. The concentration of the CCV must alternate in a repeating wave pattern: 250 µmoles, 500 µmoles, 750 µmoles, 500 µmoles, 250 µmoles, etc. On each batch, at least one 250 µmoles CCV must be analyzed to validate the curve.

A matrix spike (MS), a matrix spike duplicate (MSD), and a sample matrix duplicate must be analyzed with each batch.

When sample volume does not permit the analysis of either a sample matrix duplicate or an MS and an MSD, then a 50 µmoles/g LCSD must be analyzed to demonstrate method precision.

A second sample matrix duplicate is not routinely required unless a batch contains an NPDES sample(s) from South Carolina and the batch contains more than 10 samples.

Samples from West Virginia require a usable precision measurement for each batch. Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: an MS/MSD, a non-program deleted duplicate, or an LCSD.

See LIMS for current quality control (QC) acceptance windows. Follow guidelines in 1 P QM PRO 9015535 for outlier QC data.

Attachment:
Figure 1 (.doc)

End of document

Version history

Version	Approval	Revision information
14	06.FEB.2015	

eurofins	Always check on-line for validity. **Dilute/Run and AVS/SEM Sample Handling for Metals**	Level: **Work Instruction**
Document number: **T-MET-WI7920**		
Old Reference: **1-P-QM0WI-9013476**		
Version: **6**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **23-OCT-2014**	Document users: **6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Hg Analysis Verifiers, 6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_ICP Analysis Verifiers, 6_EUUSLA_Metals_ICP-MS Analysis, 6_EUUSLA_Metals_ICP-MS Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

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Revision Log

Revision: 6		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 P QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	Updated to include additional analysis	Added analysis 13449
Throughout document	Updated to include additional analysis	Added analysis 13449 for Non-Digest-U4
Reference	Updated to current procedure	Removed reference to SW846 6010A

Revision: 5		**Effective Date:** Apr 29, 2014
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1 P QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers with EDR numbers
Sample Collection, Preservation, and Handling	Clarification	Updated storage temperatures.

References

1. EPA-821-R-91-100, “Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment,” December 1991.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 6010B, December 1996.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 6010C, February 2007

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994

5. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471A Modified, September 1994.

6. Test Methods for Evaluating Solid Waster, SW-846 Method 7471B, February 2007.

7. Method 200.7 (rev. 4.4), *Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, USEPA 600/R-94/111 May 1994.

8. Method 200.8 (rev 5.4), *Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry*, USEPA 600/R-94/111 May 1994.

9. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020, September 1994.

10. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020A, February 2007.

11. *Chemical Hygiene Plan,* current version.

Cross Reference

Document	Document Title
Analysis #0259, 0159	Mercury in Aqueous, Solid and Tissue Samples by Cold Vapor AA
Analysis 6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue), CLP 5.2 (aqueous, solid, tissue) and EPA 200.8 (aqueous)
Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, …	Metals by Inductively Coupled Plasma Atomic Emissions Spectroscopy for SW-846 Methods 6010A/B/C (aqueous, solid, tissue), CLP 2.1(water/solid/tissue), CLP 4.0(water/solid/tissue), CLP 5.2 (water/solid/tissue) and EPA 200.7(aqueous)
1 P QM FOR 9009182	Working Instructions for Prep Solutions and Standards
1 P QM PRO 9015511	Liquid Sample Preservation
1 P QM QMA 9017309	Determining Method Detection Limits and Limits of Quantitation
1 P QM QMA 9017313	Establishing Control Limits

Purpose

This SOP is designed to provide consistent guidelines on how to handle Dilute/Run and AVS/SEM samples.

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Scope

This procedure applies to analyses performed in Environmental Sciences using ICP, ICP-MS and Hg CVAA for identification and quantitation of metallic constituents.

LOQs are based on annual statistical evaluation of laboratory data and are subject to change. The current MDLs and LOQs are maintained in the LIMS.

Limits of Quantitation are subject to change without notification.

Basic Principles

The digestion procedures normally performed in the metals group are not performed for dilute/run samples or AVS-SEM samples.

For these types of samples, information is entered for the appropriate instrument type using the information provided with the samples. Appropriate standards, check standards and interference check standards are added. Standards and samples are poured as needed to be run on the instrument.

The samples are analyzed directly on the instrument, with the exception of the spiked sample and a serial dilution of the same background sample. The spiked and serial dilution samples are prepared in either a volumetric flask or directly into graduated plastic digestion tubes used on the autosampler for instrument analysis. Any sample that requires a dilution is prepared in the same fashion.

The Quality Control requirements for accurately reviewing and verifying a sample data run are listed in Analysis #0259, 0159, Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …,and Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, ….

Definitions

1. Dilute/Run – A sample that the client has already digested and wants the laboratory to analyze or samples that the client wants analyzed without a digestion.

2. AVS/SEM – This test is used to calculate a molar ratio of Simultaneously Extracted Metals (SEM) to Acid Volatile Sulfide (AVS). The AVS/SEM extraction is performed by the water quality group. The metals group receives the extractions from the water quality group and analyzes the batch for metals (SEM)

Interferences

Not applicable.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

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Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability (DOC).

Initially, each employee performing the analysis must work with an experienced analyst for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an IDOC (Initial Demonstration of Capability). The IDOC and the DOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

A. Sample Collection and Handling

1. Aqueous samples are collected in plastic or glass containers and stored at 0° to 6°C, not frozen. Some clients may choose to send in their own containers for analysis.

2. Solid samples are collected in glass containers and stored at 0° to 6°C, not frozen.

3. Dissolved Metals: Samples to be analyzed for metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt. Samples are collected in plastic containers and preserved to a pH of <2 with HNO_3.

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B. pH Adjustment (if required):

1. Upon receipt at the laboratory, Sample Storage personnel check the pH of water samples. If the pH is greater than 2, the pH of the sample is adjusted to a pH less than 2 following the protocol outlined in 1 P QM PRO 9015511.

2. Samples requiring pH check immediately before digestion and analysis must be tested and if pH is greater than 2, the client service representative (CSR) is notified. The CSR must notify the client for direction on how to proceed with the sample (i.e. proceed as is or add more acid.)

3. Solid samples require no chemical preservation.

C. Storage - Store sample digestates in plastic bottles at room temperature. Store standards, and digestates separately.

D. Sample Discard - The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

1. Filtering apparatus

2. Class A volumetric flasks or equivalent– various sizes

3. Disposable Sterile Filter System Apparatus

Reagents and Standards

1. Nitric Acid, HNO3, Fisher, Trace Metals Grade, or equivalent; store at room temperature

2. Reagent water

3. Hydrochloric Acid, HCl Fisher Trace Metals Grade, or equivalent

Calibration

Not applicable.

Procedure

A. AVS/SEM samples

1. The Water Quality Department (Department 4029) performs AVS/SEM extraction (# 1630) and spikes the LCS, LCSD (if applicable), MS and MSD with the following:

1 mL of 1 ppm Hg

5 mL of ICP Spike 1A

5 mL of ICP Spike 1B

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2. Department 4029 delivers the samples to Department 4022 Prep lab. From this point on all steps are performed by Department 4022.

3. Allow the samples to settle, (DO NOT SHAKE).

a. If necessary, the extractions are centrifuged in 500 mL polypropylene bottles, (DO NOT SHAKE).

b. The centrifuge is located in the Organic Extraction Department (Department 4036).

4. Filtration

a. **When using the disposable sterile filter system apparatus**:

(1) Vacuum filter the extract through prefilter/0.45 membrane into the 250-ml filtrate receiver.

(2) During filtration, do not rinse solids from the bottom of the bottle. Discard solids in waste receptacle. Rinse the bottle, after solids have been discarded, with reagent water.

(3) Transfer the filtered extract from the 250-ml filtrate receiver to a 250-ml volumetric flask.

(4) Rinse the 250-ml filtrate receiver. Transfer the rinsate to the 250-ml flask. Dilute to volume with reagent water and mix well.

(5) Transfer the mixed sample back into the original reagent-washed 500-ml polypropylene (PP) bottle. Take to ICP or ICP-MS and notify one of the analysts that the samples are ready for analysis.

b. **When filtering into a flask:**

(1) Vacuum filter the extract through prefilter/0.45 membrane into filter flask.

(2) During filtration, do not rinse solids from the bottom of the bottle; discard solids in waste receptacle. Rinse the bottle, after the solids have been discarded, with reagent water.

(3) Transfer the filtered extract from the filter flask to a 250 mL volumetric flask.

(4) Rinse the filtering flask and transfer the rinsate to the 250 mL flask. Dilute to volume with reagent water and mix well.

(5) Transfer the mixed sample back into the original reagent washed 500 mL polypropylene (PP) bottle. Take to ICP or ICP-MS and notify one of the analysts that the samples are ready for analysis.

5. Once the samples have been delivered to ICP or ICP-MS for analysis, an analyst assigns a batch number and transfers the information into the departmental LLENS system which is uploaded to parallax.

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6. If the samples require Hg, the Hg analyst is informed that the samples are ready and given the batch number that is assigned to the samples.

7. The samples are then ready to analyze on the instrument.

8. The samples follow the same routine as any other samples that are analyzed on the instrument.

B. Dilute/Run samples – “As Received” samples

1. The client sends the sample into the lab.

2. The prep scheduler generates a #4792 (for SW846 update 3) or a #13449 (for SW846 update 4) list to determine what samples need to be analyzed.

3. The analyst requisitions the samples in Parallax.

4. The analyst retrieves the samples from storage at the requested time.

5. The analyst batches 20 or less samples per analytical batch.

6. The analyst makes a PBW, LCSW and LCSDW. See 1 P QM FOR 9009182 for additional information.

7. The analyst then documents the batch number, LCSW and LCSWD spiking information, PBW and all sample numbers included with this batch in the departmental LLENS system which is uploaded to parallax.

8. The samples are ready to analyze on the instrument.

9. The samples then follow the same routine as any other samples that are analyzed on the instrument.

Calculations

1. Final Result

a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

All dilution factors must be recorded and used in the calculation. [To enter dilution data into the LIMS when multiple dilutions are used, a factor must be formed (Ex. 1), which contains no more than three figures for the volume or the aliquot (Ex. 2).]

Ex. 1. $50/.5 \times 10/1 = 500/.5$

Ex. 2. 50/.5 × 25/.5 = 1250/.25 = 125/.025

NOTE: The default units are µg/L

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{(\sum X^2 - \frac{(\sum X)^2}{N})(\sum Y^2 - \frac{(\sum Y)^2}{N})}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\%\ Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

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SR = sample result

6. Methods of standard additions (MSA)

Take 4 identical aliquots of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution. Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ≥0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

Statistical Information/Method Performance
Please consult 1 P QM QMA 9017309 and 1 P QM QMA 9017313.

Quality Assurance/Quality Control
Please consult Analysis #0259, 0159, Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …, and Analysis #6966, 1643, 6935, 7914, 6946, 6947, 1650, 6949, 6952, 6951, 6953, 1654, 1662, 1656, 1657, 6958, 6960, 1667, 6961,10145, 6955, 6944, 6936, 6969, 7968, … for specific QC protocols and procedures.

End of document

Version history

Version	Approval	Revision information
6	23.OCT.2014	

eurofins	Always check on-line for validity.	Level:
Document number: **T-MET-WI11931**	**Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)**	**Work Instruction**
Old Reference: **1-P-QM-WI-9018442**		
Version: **14**		Organisation level: **5-Sub-BU**
Approved by: **UDM6** Effective Date **30-NOV-2020**	Document users: **6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_ICP Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

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Appendix I

Revision Log

Revision: 14	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Quality Assurance/Quality Control	Current Practice	Updated EPA batch size to 20 samples
Reference	Current Practice	Added new verification SOP
Routine Methods	Current Practice	Removed Analysis numbers from previous system
Apparatus and Equipment	Current Practice	Removed products not being used anymore
Reagents and Standards	Current Practice	Updated to current Definitions
Throughout Document	Current Practice	Updated SOPs to D4 document numbers
Procedure	Current Practice	Updated procedure to new LIMs operation
Instrument Operations	Current Practice	Updated to new LIMs operation
Routine Maintenance for ICAP	Current Practice	Updated to better practice and more detail
Calculations	Clarification	added units to Water sample and added LIMs ID to calculations

Revision: 13	Effective Date:	17-JUL-2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Table II	Correction	LLC windows are 50-150%, removed 20% WW on LCS

Reference

1. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010B, December 1996.

2. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010C, February 2007.

3. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010D, Rev.4, July 2014.

4. Method 200.7 (rev. 4.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, USEPA 600/R-94/111 May 1994.

5. ICAP™ 6000 Series ICP-OES Spectrometer Operator Manual, 2005/2006.

6. *Hardness Calculation SM 2340 B.*

7. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10695.	Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
T-MET-FRM9070	MSA Prep for Samples with MS/MSD <50%
T-MET-FRM9072	MSA Prep for Samples within TCLP Limits
T-MET-WI12063	ICP Solutions and Standards Preparations
T-MET-WI9082	Working Instructions for Prep Solutions and Standards
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
T-MET-WI35797	Metals Data Verification Process

Scope

This procedure applies to analyses performed in the Metals department using Inductively Coupled Plasma (ICP) Atomic Emissions Spectroscopy for identification and quantitation of metallic constituents by Methods 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous).

The limits of quantitation (LOQ) are based on annual statistical evaluation of laboratory data and are subject to change without notification. The current method detection limits (MDLs) and LOQs are maintained in the laboratory information management system (LIMS).

This SOP also outlines the proper operation and maintenance of the ICP instrumentation and provides consistent guidelines for the evaluation of ICP data.

Routine Methods

Elements routinely analyzed on the Thermo Scientific iCAP 6000 and 7000 Series Analyzers:

Element	Wavelength (nm)
Ag	328.06
Al	308.21
As	189.04
B	249.67
Ba	455.4
Be	313.04

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Ca	317.93
Cd	226.5
Co	228.62
Cr	267.72
Cu	327.4
Fe	261.19
K	766.49
Li	670.78
Mg	285.21
Mn	257.61
Mo	202.03
Na	589.59
Ni	231.6
P	177.49
Pb	220.35
S	182.03
Sb	206.83
Se	196.09
Si	251.6
Sn	189.99
Sr	421.55
Th	401.913
Ti	334.94
Tl	190.86
V	292.4
W	207.911
Zn	213.86
Zr	339.19

Basic Principles

If required, samples are digested with acids and heated to solubilize metals. Samples are received from the prep area in digestion vials.
After the analytical batch is prepared it is loaded onto the autosampler for trace metals analysis.

Samples are transported to a nebulizer via an autosampler and peristaltic pump. The nebulizer introduces an aerosol into a spray chamber; the resulting mist is then transported to an argon plasma torch where excitation of atoms occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency (R.F.) inductively coupled plasma. The spectra are dispersed by a diffraction grating and the intensities of the light at each wavelength are monitored by a photosensitive device. The signals from the photosensitive device are processed by a computer. A background correction technique is required to compensate for variable background contribution to the spectra of trace elements.

Interferences

Spectral interferences are caused by background emission, stray light from high concentration elements or overlap from a spectral line from another element. Spectral interferences are compensated for by the use of background points, alternate wavelengths and interelement corrections.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for by using internal standardization.

Memory interference, or carryover, is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the autosampler tubing overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

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Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCI with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Fill reagent bottles in an operating fume hood using caution to avoid spills.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an IDOC (Initial Demonstration of Capability).

The IDOC consists of four laboratory control samples (LCS) that are carried through all steps of the prep and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

A. Preservation and Holding time

1. All samples are typically shipped and stored at 0°to 6°C, not frozen, but room temperature is acceptable. Samples are analyzed within 6 months of collection.

2. Aqueous samples are collected in plastic or glass containers and are preserved to pH <2 with nitric acid. If samples are received > pH 2, they are preserved at the lab and held at least 24 hours until digestion. See *S-SS-WI10695*.

3. Drinking water samples are digested if the turbidity is > 1 NTU. See *S-SS-WI10695*.

4. Dissolved metals must be filtered within 15 minutes of sampling. If this is not done, the lab filters the samples of unpreserved bottles within 5 days of submission to the laboratory. Lab-filtered samples are preserved to pH <2 with nitric acid after filtration.

5. Solid samples are collected in glass containers with no chemical preservation.

6. Sample digestates are stored in plastic bottles at room temperature.

B. Sample Discard - The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

The following is a list of the hardware and apparatus necessary for ICP analysis. More detailed hardware information is located in the *Operator's Manuals*.

1. 15-mL graduated polypropylene tubes with caps (certified ±1%)

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2. FilterMate filtration device with 0.45-µm PTFE fiber filter and insertion tool

3. 10-mL sterile disposable syringes

4. syringe filters, PTFE, 0.45-µm

5. 30-mL polypropylene medicine cups

6. Calibrated hand-held pipettes and tips (10 – 5000 µL) - FisherBrand or equivalent.

7. Spectrometer - The Thermo Scientific iCAP™ 6000, 6500, and 7000 series Trace Analyzer utilizes a high performance solid state Charge Injection Device (CID) camera system to deliver high contrast/low noise imaging and quantification of all wavelengths in the analytical range. With the entire spectrometer and foreoptics purged with either Argon or Nitrogen, it features a 52.91 grooves/mm grating and dual-view detector.

8. Auto-sampler – The ESI SC-14 auto-sampler and integrated "FAST" system offer increased capacity and reduce sample introduction times. The parameters for each automated run are entered into the auto-sampler table in the iTEVA™ software as described in Instrument Operation, Section A of this SOP.

9. Peristaltic pump – The peristaltic pump regulates the flow of the following: sample, internal standard, instrument rinse and spray chamber waste. Special care must be taken to ensure that all pump tubing is connected properly. The Teflon concentric and glass V-groove nebulizers have a natural uptake, but a peristaltic pump is used to compensate for differences in sample viscosity. After traveling through the peristaltic pump, the sample and internal standard tubing are combined by a "T" connector, and then allowed to mix in a mixing coil before entering the nebulizer.

10. R.F. generator –The iCAP™ 6000 series Trace Analyzer utilizes an internal solid state RF Generator at 27.12MHz with a power efficiency greater than 78%.

11. Coolflow –The ThermoFlex™ 900 cooling device for the iCAP™ 6000 series Trace Analyzer operates at 17°C.

12. Personal computer – The iCAP™ 6000 series Trace Analyzer is controlled by PCs.

Reagents and Standards

Reagent and standard information and the preparation of the following standard and solutions are located in *T-MET-WI12063*:

Initial Calibration (ICAL)
Blanks and calibration standard
Initial Calibration Verification (ICV) standard
Continuing Calibration Verification (CCV) standard
Rinse/Carrier and Profile Solutions
Interference Check Solutions (ICSA and ICSAB)
Low Level Check (CRI) standards
Post Digestion Spike (PDS)
Serial Dilution (SD)
Linear Range Standards (LRC)
Internal Standard Solution
Instrument Detection Limit (IDL) and MDL Solutions

Calibration

See T-MET-WI35797 for the frequency, acceptance criteria and corrective action of the ICAL, ICV, ICB, CRI, ICSA/ICSAB.

Procedure

A. Setting up an ICP run sequence file

1. Determine the batches to be analyzed and determine any special requirements by viewing lab notes and/or project notes that are with the batch paperwork delivered from the metals prep area.

2. Assign an ICP metals storage location and document the location on the batch sheet(s) and on the sample lid for the Prep Blank of each batch.

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3. Complete necessary pouring for the prep batch following Section B

a. Batches with only field blanks or equipment blanks do not need a post-digest spike or a serial dilution.

b. "As Received" samples must be run with a blank and LCS, LCSD (prepared and documented in the LIMs system by the analyst).

4. Open run template following Instrument Operations Section 2

5. Scan samples by digestate bottle into program

6. When setting up a run, Batch QC must be placed in the same block of ten or fewer samples. If there are two LCSs, they must be placed one after the other. [The order of the batch QC is typically MB, LCS, (LCSD), BKG, PDS, DUP, MS, (MSD) and SD.]

a. ICV/ICB must be analyzed immediately after the calibration curve.

b. CRI, ICSA, ICSAB, CCV, CCB must immediately follow the ICV/ICB.

c. 6010D requires analysis of LRCs at the beginning of the analytical run

c. CCV/CCB must be analyzed after every ten analytical samples.

d. Additional QC are added per client requirements at the end of the run: CRI, ICSA, ICSAB.

e. Any deviations from protocol must be noted in the LIMs system

7. Edit dilution factors (DF) and auto-sampler locations as needed.

8. Remove all unnecessary QC standards by selecting individual lines and clicking on the red "X" button.

9. Save the sequence file

B. Pouring ICP samples and QC samples

It is important to minimize any chance of contamination, both of yourself and the samples. Keep your hands and the work area clean at all times. Do not re-use any pipette tips.

Run QC standards are prepared separately, and kept in separate auto-sampler racks to be obtained at the time of analysis.

1. Use the prep paperwork to document the following information when pouring samples: initials, employee number, PDS lot number, and the date.

2. Carefully examine the batch to ensure there are no discrepancies between the Batch Preparation Sheet and the physical placement of samples in the batch.

3. Obtain and label all required test tubes, and place them in test tube racks. Any poured sample that does not require a graduated test tube is to be poured into a polystyrene tube.

4. Prepare and label the PDS required for each new batch (sample volume permitting).

a. A PDS is prepared using 0.2 mL of a custom-ordered PDS solution into 9.8 mL of background sample.

i. If sample volume is limited, reduce volumes but keep ratios equivalent (i.e. 0.1 mL PDS solution into 4.9 mL background sample.)

ii. If the background sample must be diluted, add a reduced volume of background sample and bring to volume with the appropriate matrix.

5. Prepare a serial dilution (SD) of the background sample with a DF5 by pipetting 2 mL of background sample into 8 mL of the appropriate matrix.

a. If the background sample chosen for serial dilution has been diluted due to matrix interference or to bring the concentration into the linear range of the instrument, the serial dilution must also be diluted by 5 times the dilution factor of the background (i.e., if Bkg = DF5, S.D.

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must = DF25).

6. If samples need to be filtered due to cloudiness or contain particulate, then the corresponding method blank (MB) and laboratory control sample (LCS) must be filtered.

a. Filtered using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.

c. Document lot of filters on the preparation batch sheet and mark each sample lid with an "F" to indicate that it was filtered.

7. For TCLP samples that requires method of standard additions, refer to *T-MET-FRM9072* or *T-MET-FRM9070* for instructions.

8. Perform any additional spiking or dilutions and document on the preparation batch sheet

9. Cover the samples with lids or with plastic wrap to prevent contamination.

10. Place the poured batch on the bench top to await analysis, or return samples to their ICP sample storage location.

11. Take **NOTE** of the following:

a. A PDS and a serial dilution (SD) must be performed on one sample in each digestion batch.

b. Analysis information, including standard lot numbers, run number, method, analyst and date of analysis, are captured in the LIMs system

c. Documentation is of utmost importance. Double check all entries.

d. Dilute samples when necessary to yield a response that falls within the calibration range. When there are multiple analytes over range, only run a maximum of three tubes, the DF 1 if needed, the highest dilution required for the highest analyte, and a dilution in between.
i) Exception: If analytes are over range at a DF1, and the next dilution they are ND, take the data

e. If a batch requires re-analysis, it is acceptable to re-use dilutions and/or spiked samples that were prepared for the previous analysis. A "P" on the preparation batch sheet is used to indicate that a previously poured test tube is being re-used.

Instrument Operations

A. iTEVA™ software: The Thermo Scientific iCAP instrument is operated through iTEVA™ software. From the Start menu, select "iTEVA™ Control Center" to start the software.

1. Plasma ignition

a. Open the plasma status window by clicking on the icon at the bottom of the screen.

b. Verify that all parameters are within acceptable range for ignition as indicated by a green light.

c. Ensure that the drain tubing for the spray chamber is properly connected to the peristaltic pump and positioned to drain into a waste carboy.

d. Select "Ignite plasma". After the plasma has ignited, the instrument automatically performs optimization of the nebulizer gas pressure, and then starts the on-board peristaltic pump.

e. If the plasma does not light, repeat steps b-d.

f. Once the plasma operating parameters have engaged, exit the Plasma status window by clicking on Close.

g. If the plasma has been off for more than 15 minutes let the instrument warm up for 30 minutes. If the plasma has been off less than 15 minutes, let the instrument warm up 5 to 10 minutes.

2. Automated analysis using the iTEVA™ software

a. Open the Analyst window from the Control Center by clicking on the Analyst icon. When prompted, select the appropriate method.

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b. In the Analyst window, click on the Sequence tab at the bottom of the screen.

c. From the Auto-session menu, select New Autosampler.

d. The “New automation-session” window opens.

e. Click on “New” to add a sequence and the “New sequence” window appears.

f. Click on the “Import from delimited text file” option and choose an appropriate template or previously prepared run sequence from the drop-down menu for the run to be entered.

g. Change the sequence name to reflect the run number, but do not change the method revision number in the parentheses.

h. Click OK at the bottom of the New Automation session window to continue.

i. The sequence template is loaded. When prompted in the import warning window, click OK to choose the populate the auto-sampler.

j. Right-click on the most recent auto-sampler session and select “Auto-locate All” to have autosampler positions automatically assigned.

k. Click on the "list view" icon and scan calibration information into the table using barcodes provided.

l. Scan client samples into table from digestate bottle to include sample number and hand enter the dilution factor if greater than a DF1.

l. Additional samples and QC are added using the “Add sample” and “Add QC” icons, respectively, at the top of the screen. Unused rows are removed by selecting the entire row(s) and using the “remove sample” icon at the top of the screen.

m. Click on the printer icon and choose to print page 1 only. This printout is kept in the designated bin in ICP office. Document on the print out the prep batch and analytical batch number.

n. Verify that the sample list begins and ends at the correct tube numbers, and check all entries for errors.

o. Click on the “Initialize Autosampler” icon to initialize the autosampler.

p. Carefully examine the batch to ensure there are no discrepancies between the preparation batch sheet, auto-sampler sequence table and the physical placement of samples in the batch. Sign off on the sequence table sheet to indicate these items have been checked.

q. Click on the “Run auto-session” icon to start the run sequence. If a run sequence is to be started at a sample or standard other than the initial calibration, open auto session run using the plus sign. Open the run and use the plus sign to open the required sample or standards. Right-click on the appropriate sample or standard and choose “Start Auto-Session Run at this Sample”. Deselect the reslope (or Calibration option if running Multi Point Method) and QC, then click OK.

3. Running an AutoPeak in iTEVA™

a. In an open autosampler session (autosampler must be initialized), right-click on the location of the AutoPeak solution, and choose “Go to...”

b. Click on the Analysis tab to open the Analysis window.

c. From the Instrument menu, select Perform AutoPeak.

d. Click on All elements, then OK.

e. When prompted to aspirate the high standard, click OK. The AutoPeak is now performed.

f. When the AutoPeak has finished, click on Done.

4. Manual analysis in iTEVA™

a. In an open autosampler session (autosampler must be initialized), add sample(s) to be analyzed to a new or existing sequence as detailed in section A.2 of this section.

b. To start analysis, right-click on the appropriate sample and choose “Start Auto-Session Run at

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this Sample" and elect not to run the "start actions."

c. When analysis is complete, results can be printed from the Analysis tab by right-clicking on the sample name and selecting "Print Sample…"

5. Performing an interelement correction (IEC) in iTEVA™

a. Prepare a solution of the interfering element at the linear range of the instrument. Perform manual analysis according to section A.4 of this section.

b. Note results of elements with a known interference that are greater than the limit of quantitation.

c. Divide the result of each interfered element by that of the interfering element. These values represent the amount the current IECs need to be adjusted.

d. In the Method window, click on Elements to expand the list of elements in the method.

e. Click on the targeted element to view the settings for that element.

f. Click on the IECs tab to view the current IECs for that element.

g. Add each calculated result to the correction factors currently in the first column corresponding to each pair of elements.

h. Enter the new correction factors in the table.

i. Repeat steps d-f as needed.

j. Click on the Save method icon to save the method.

k. For routine IECs that are completed every six months, analyst updates the LIMs database with the information provided by the analyst. For non-routine IECs the analyst must document changes in the instrument log book with updated information.

B. Import Analytical and Raw data into LIMs System

1. Right click on Run Number and select Export All Samples

2. Enter the run number, copy the run number, and hit OK.

3. Go into the IDAT-Sequence folder and copy file to TALs Import folder

4. Go into TALs and find the analytical batch and obtain the designated analytical run number determined by the LIMs system

5. Go into the IDAT-Sequence folder and print text file as PDF and name PDF the same as the analytical batch

6. Sign into ShareFile

7. Go to the Personal Folders and hit the + button to add file and choose UPLOAD

8. Browse files

9. Find the analytical batch file in the IDAT Sequence folder and select Open

10. Upload file. This will now be in analyst personal Share File

11. Open TALs and open the analytical batch that is currently being processed

12. Select DOCs button on the top of the analytical batch

13. Right click in the box that pops up and choose Add New Document

14. Find your Citrix Files on the right hand side to access the Personal Folder

15. Double click on the Personal Folder to open it up to access the raw data file and choose Open

16. Click on the file and hit Select and then hit OK

17. Raw data is now attached

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Routine Maintenance for the ICAP™ 6000 Duo Analyzer

A. Sample introduction system removal and cleaning: Remove and clean the sample introduction system when instrument performance declines (See **Figure 1**). Any adjustment to an instrument (replacement of parts, etc.) must be documented in the appropriate instrument logbook.

1. Turn plasma off and let instrument cool for a minute

2. Remove spray chamber and nebulizer.

   a. Unclamp spray chamber from spray chamber adapter by gently squeezing clamp with one hand while supporting the spray chamber with the other hand.

   b. Gently remove the nebulizer from the spray chamber by pulling it out.

   c. Disconnect the argon and sample tubing from the nebulizer by pinching the Luer lock and pulling the tubing off the nebulizer.

   d. Clean the spray chamber if residue is observed coating the sides. If cleaning is necessary, remove the drain tubing from the spray chamber.

3. Remove and disassemble the torch

   a. Gently pull the spray chamber adapter out of the torch assembly.

   b. Unlock the torch assembly by turning it clockwise and remove it from the instrument.

   c. Turn the injector tip assembly counter clockwise to separate it from the torch housing.

   d. Gently pull the center tube out of housing to clean.

4. Prepare ultrasonic bath. Make sure the bath is at least ½ full with clean reagent water.

5. Clean radial cup

a. remove gently from box by wiggling back and forth
b. place radial cup in medicine cup and fill inside radial cup and medicine cup with matrix rinse
c. allow it to soak for 10 minutes and then rinse off with reagent water
d. dry radial cup and place back in torch box prior to reconnecting the torch

5. Clean torch, injector tip, and nebulizer.

   a. Invert the torch in a 250-mL vacuum flask of 50% HCl and place in the sonicator for 10 minutes. After sonication, rinse the torch with reagent water. (Be careful not to get a lot of water down into the base of the torch.) Carefully dry the torch with a paper towel.

   b. Place the injector tip in 50% HCl for 10 minutes. Rinse the injector tip with reagent water and carefully dry with a paper towel.

   c. For a plastic nebulizer, use a syringe to force the 50% HCl solution through the nebulizer to clean and/or remove any clogs.

   d. If the spray chamber needs to be cleaned, place it in the sonicator for approximately 5 minutes, and then rinse it out with matrix rinse, followed by reagent water.

B. Reassemble the sample introduction system: (See **Figure 1**)

1. Reassemble the torch.

   a. The o-rings on the metal torch mount must be inspected and replaced if any wear or damage is visible.

   b. The quartz torch is pushed fully into the metal torch mount with a gently twisting pressure.

   c. The circular "target" design on the torch **MUST** align with the circular notch on the torch mount.

   d. Insert the injector tip fully into the center tube holder.

   e. Insert the center tube assembly into the torch mount and rotate it counter-clockwise to lock it

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in position.

f. Mount the torch assembly back into the instrument by inserting it straight through the torch hole and coil, being careful not to disturb the quartz bonnet above the radial view lens.

g. Turn the assembly counter-clockwise to lock it into position.

h. Gently push the spray chamber adapter into the back of the center tube assembly.

2. Reassemble the spray chamber and nebulizer.

a. If removed for spray chamber cleaning, reattach the drain tube to the spray chamber.

b. With a twisting motion, insert the nebulizer into the spray chamber so that the collar is a tight fit.

c. Attach the sample and nebulizer gas tubing to the nebulizer.

d. Clamp the spray chamber to the spray chamber adapter.

3. Ignite the plasma.

C. Changing the pump tubing: Change pump tubing on the peristaltic pump when the tubing shows wear. Inspect all tubing to insure that it is secure and in good condition.

D. Documentation for instrument/analysis tag out and return to service.

NOTE: The following information is taken from QA-SOP11901: In the event of an equipment failure, the following must be performed:

1. Document the nature of the failure in the maintenance logbook

2. Document how and when the defect was discovered

3. Notification of supervisor or responsible person who can decide on appropriate action to take

4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:

a. Date taken out of service

b. Employee who took the instrument out of service

c. Reason for tagout

Form Q-EQA-FRM6871is used for "tagging out".

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken to bring the equipment back into service.

7. Results of the corrective action (i.e., system calibration within specifications, etc.)

8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This is done on a case-by-case basis.

9. After repair, document whether the function has been fixed. Then determine if calibration or verification activities need to be performed before the instrumentation is put back into service.

Calculations

1. Final Result

a. Water sample (mg/L)

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$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

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$$\% \ Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)
Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or 2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y-intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ³0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

7. Total Hardness Calculation (SM2340B), Standard Methods 2340B-2011. The calcium result (mg/l) is multiplied by a factor of 2.497, the magnesium result is multiplied by a factor of 4.118, and the two values are added together for the hardness result.

8. Trivalent Chromium (7199_CR3) water and soils. Calculation is performed by subtracting hexavalent chromium from total chromium.

9. Ferric Iron (3500_F+3_B_Calc) is calculated by subtracting ferrous iron from the total iron.

Statistical Information/Method Performance

Generate MDLs and LOQs according to *QA-SOP11892*. Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire digestion and analysis procedure. Compare and pool results to determine the final reporting MDL. The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

QC acceptance limits (MS, MSD, LCS and LCSD) are established as statistical limits (see *QA-SOP11896* for guidance). Limits are evaluated every 6 months by the department and updated in LIMS by QA as directed by the department supervisor.

Quality Assurance/Quality Control

A. For 6010B, 6010C, and 6010D each digestion batch (up to 20 samples) must contain a method blank (MB), LCS, unspiked sample (BKG), Duplicate (DUP), MS, and MSD. If there is not sufficient sample or per client requirement and LCSD is included.

B. For 200.7, each digestion batch (up to 10 samples) must contain a MB, LCS, BKG, DUP, and MS. If there is not sufficient sample or per client requirement and LCSD is included.

C. Batch Quality Control - For the preparation and concentrations see *T-MET-WI12063* and for the frequency, acceptance criteria and corrective action see SOP T-MET-WI35797.

D. Data Quality Checks

1) For SPLP and TCLP samples, an MSA (method of standard additions) is required if:

a. The sample concentration falls between 80% to 100% of the regulatory limits.

b. If the SPLP or TCLP Matrix Spike (QA) recovers < 20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

2) For all EW samples (samples from public drinking water sources); check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed in QA-SOP11886. Suppliers must be notified within 24 hours.

3) For the iCAP 6000 series instrument (T70, #11016; T71, #16315; T72, #16417, T73, #18255 , T75#27278) Si is monitored due to an interference on Pb. Pb data must be reread if Si is not within ±10% in CCVs.

F. When raw data checks are complete, check the following:

1. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms. Any dilutions required have been calculated correctly and added to the reread/redigestion form. Specific instrument has been noted for client requirements if necessary.

2. Data for samples following Good Laboratory Practices (GLP) must be retained as permanent storage.

G. Instrument detection limits are performed when an instruments detector is changed or a new instrument is added to the lab. Method detection limits (MDLs) follow guidelines in SOP ***QA-SOP11892***. Inter-Element Correction factors (IECs) are checked routinely every 6 months for each analytical instrument. Linear ranges are verified semi-annually.

H. For data review reference SOP T-MET-WI35797

Appendix I

Definitions and explanations of the codes and symbols used on the raw data

A. Sample table information

1. The run number.

2. The page number.

3. The tube number.

4. The sample number.

5. The first and second of four asterisks denote whether the sample is a background (U*), duplicate

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(D*), spike (R*), MSD (M*), post-digestion spike (UP), serial dilution (UL), or not a QC sample (**).

6. The weight to volume or volume to volume digestion ratio, consisting of the initial quantity of sample used and the final digest volume.

7. The dilution factor – Indicating if the digest solution was diluted prior to analysis. An undiluted sample is labeled DF1.

8. Digestion batch number – Assigned when designated samples are scheduled for preparation, this number is used to track samples and QC prepared together.

9. The protocol by which the data is reviewed (SW-846, EPA-600).

10. Date and time of the sample injection into the instrument.

11. The ICAP identification number.

B. The ICP scans all of the method elements simultaneously during the analysis. The QC review lists all the samples on the run. The QC review lists elements verified, good phantom, and elements/phantom that are bad (need to be reread for run or batch QC). The reviewer or verifier documents on the QC review if any element(s)/sample(s) were selected/deselected.

C. The following are error codes in the iTEVA™ software.

1. S = Saturation – The concentration of the element is more than the detector can quantify.

2. K = The Elements Affected by a Saturated Element – The concentration listed is not accurate, and a more accurate result can be obtained by running the sample at a dilution.

D. Along with the average concentration (in ppm), the average intensity, %RSD and all three replicates are shown for each analyte. Internal standard values are intensities (cts/s).

Attachment:
Figure 1 (.doc)

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
S-SS-WI10695 Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
T-MET-FRM8802 ICP LOQs (mg/L)
T-MET-FRM9070 MSA Prep for Samples with MS/MSD <50%
T-MET-FRM9072 MSA Prep for Samples within TCLP Limits
T-MET-WI12063 ICP Solutions and Standards Preparation
T-MET-WI9082 Working Instructions for Prep Solutions and Standards

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End of document

Version history

Version	Approval	Revision information
12	05.MAR.2019	
13	12.JUL.2019	
14	23.NOV.2020	

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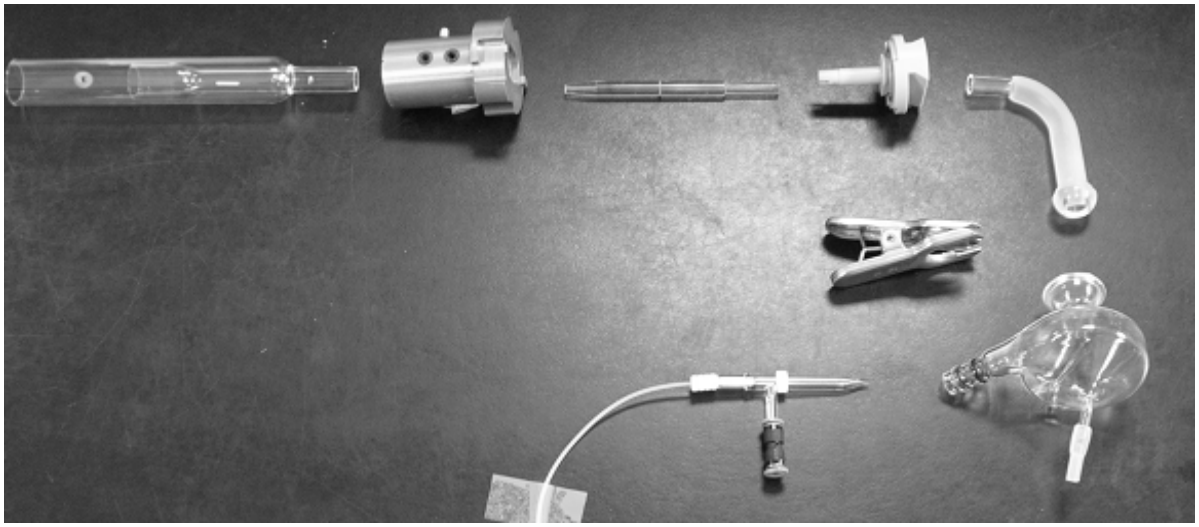
Figure 1



KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-551
Revision History
Cover Page
Page 1

TITLE: GRAIN SIZE ANALYSIS

Prepared By: Jessica Spurlin Date: 10-1-15

Approved By:

Department Manager: [illegible] Date: 10-1-15

Operations Manager: Deborah J. Radeau 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
03	Sect. 4 & 7 - Added wooden tongue depressors and removed "rubber-tipped" from pestle. Updated references	LAD	06/20	06/20

TITLE: **Grain Size 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-551-03,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-551-03,** titled **GRAIN SIZE ANALYSIS.**

Recipient: _____Date:_____

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 Pestle

4.13 Glass beakers- 1000ml and 500ml

4.14 Wooden Tongue Depressors

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 characteristics for each sample, determine 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 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.

7.4.3 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.

<u>Large Sieves</u>

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.

<u>Small Sieves</u>

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 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)

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)

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not Applicable

TITLE: **Grain Size Analysis**

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 (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

LIST OF TABLES AND FIGURES

Table 1 Percent Solids Table for Weight Determination
Table 2 Summary of Method Modifications
Figure 1 Example of Logbook Page

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	Hydrometer 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	Hydrometer 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

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-551-03	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:	Mafic/organics - removed 3.1g sticks/grass/roots		
Sample Weight	Sample (g)	Date/Time in oven	See pg. 1
Sample Weight (wet)	~~1250~~ 89.9	Date/Time out of oven	
Sample Weight (dried)	25.98		
		Hydrometer Data	
% Moisture	71.05	Serial Number	742303
		Cal Date:	3-25-19: 11:32
Sample Split (Oven Dried)	Sample (g)	Low Temp C	16.4
Sample >=#10	Ø	Low Temp Reading	1.0035
Sample <=#10	25.98	High Temp	17.6
		High Temp Reading	1.0035
Sieve Only:			
Sieve Only, Wet Wash:			
		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	270.0	270.6
#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	340.3

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)	17.3	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

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-777
Revision History
Cover Page
Page 1

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

Prepared By: George Brewer Date: 05/22/13

Approved By:

Department Manager: George Brewer Date: 05/22/13

Operations Manager: Deborah J. Radeau Date: 5.22.13

QA Officer: Leslie Dimond Date: 05.22.13

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK 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-777-00**, titled **TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD**

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-777-00**, titled **TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD**

Recipient: _____Date:_____

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure and requirements followed by Katahdin Analytical Services, Inc. for the titrimetric determination of organic carbon. This method is applicable to the measurement of carbon in soils and sediments in accordance with the method of Walkley and Black. The minimum reporting limit for this method is 0.1% on a dry weight basis.

1.1 Definitions

Laboratory Duplicate – a second aliquot of a sample that is analyzed 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.

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 titrimetric analysis of organic carbon by the Walkley-Black method. 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 titrimetric analysis of organic carbon by the Walkley-Black 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.

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

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 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; not taking food or drink into the laboratory; and knowing 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.

Any residual basic waste generated from the preparation of reactive organic carbon in this analysis is placed in satellite “G” or pyridine waste. The acidic waste after titration is put in satellite “A” or acid waste.

Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin 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

An aliquot of a soil sample is oven-dried, ground, and sieved prior to analysis. Organic carbon in the sample is oxidized with a measured excess of potassium dichromate in the presence of sulfuric acid. Dichromate ion reacts with organic carbon as follows:

$$2Cr_2O_7^{2-} + 3C^0 + 16H^+ \rightarrow 4Cr^{3+} + 3CO_2 + 8H_2O$$

Excess dichromate ion is then back-titrated with ferrous iron. Ferrous iron reacts with dichromate as follows:

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

The endpoint of the titration is indicated by the formation of a red-brown complex between excess ferrous iron and o-phenanthroline using ferroin indicator.

3.0 INTERFERENCES

3.1 High chloride concentrations will give a positive error.

3.2 Ferrous iron in the sample will give a positive error. Drying the sample prior to analysis in the presence of air oxidizes ferrous iron to ferric iron.

3.3 The method will not measure element organic carbon, such as soot carbon or charcoal.

4.0 APPARATUS AND MATERIALS

4.1. Buret, 50 mL, graduated to 0.1 mL (Class A)

4.2. Stir plate

4.3. Buret stand and holder

4.4. Mortar and pestle, porcelain or glass

4.5. Brass sieve, 0.5 mm

4.6. Top loading balance readable to 0.001 g

4.7. Stir bar and retriever

4.8. 500 mL Erlenmeyer flask

4.9. Adjustable automatic pipets or volumetric pipets-10mL, 1 mL

4.10. Volumetric flasks, Type A

5.0 REAGENTS

5.1 Laboratory Grade Reagent Water: organic carbon-free water.

5.2 Potassium dichromate ($K_2Cr_2O_7$) solution, 1N: Dry ACS reagent grade potassium dichromate in an oven at 103°C – 105°C overnight, and store in a dessicator until use. Dissolve 49.04 g of potassium dichromate in water in a 1000 mL volumetric flask, and

bring to a final volume of 1000 mL. Store in a HDPE bottle. Prepare fresh every 6 months.

5.3 Sulfuric acid, concentrate, ACS reagent grade

5.4 Ferroin indicator, purchased.

5.5 Potassium hydrogen phthalate or KHP, acidimetric standard, ACS reagent grade, oven dried at 103^oC – 105°.

5.6 Organic carbon standard, 10000 mg/L: Dissolve 2.128 g oven-dried KHP in reagent water in a 100 mL volumetric flask. Dilute to volume and store in a glass amber bottle at 4 +/- 2^oC for up to 6 months.

5.7 Ferrous sulfate heptahydrate ($FeSO_4 \bullet 7H_2O$) titrant: In a 1000 mL volumetric flask, dissolve 140 g ACS reagent grade $FeSO_4 \bullet 7H_2O$ in approximately 500 mL reagent grade water. Add 15 mL of concentrated sulfuric acid. Cool and dilute to volume with reagent grade water.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Samples should be taken in glass or plastic bottles, and stored at 4 +/- 2^oC until analysis.

6.2 Samples must be analyzed within 28 days from sample collection.

7.0 PROCEDURES

7.1 SAMPLE PREPARATION

7.1.1 Samples are dried at 103° C – 105° C prior to analysis. Sufficient sample should be prepared to provide a 10 g aliquot for analysis.

7.1.2 Dried samples are ground using a mortar and pestle to as fine a powder as possible.

7.1.3 The ground sample is passed through a 0.5 mm brass sieve, and the sieved sample is subsampled for analysis.

7.2 STANDARDIZATION OF FERROUS SULFATE TITRANT

7.2.1 Using a volumetric pipet, add 10.0 mL of 1N potassium dichromate solution to a 500 mL Erlenmeyer flask.

7.2.2 Using a bottle-top dispenser, carefully add 20 mL of concentrated H_2SO_4 to the flask. Perform this operation in a fume hood. Swirl the flask to mix the reagents and allow to cool in the fume hood for 30 minutes.

7.2.3 Using a graduated plastic measuring cup, add 200 mL of reagent water to the reagents in the flask and swirl to mix.

7.2.4 Add 5 to 10 drops of ferroin indicator to the flask and place a Teflon stir bar to the flask. Stir the mixture continuously on a stir plate while titrating with the ferrous sulfate titrant. The mixture will turn olive green, then deep green, and then green-blue as the endpoint is approached. When the end point is near, add titrant dropwise until the color turns reddish-brown at the endpoint. This color change is very sharp.

7.2.5 Record the volume of titrant used to the nearest 0.05 mL.

7.2.6 Calculate the normality of the ferrous sulfate titrant using the following equation:

Ferrous Sulfate Normality = 10 / (mL of titrant)

7.2.7 If the titrant normality is not 0.500 +/- 0.05N, remake the titrant and the potassium dichromate solution and retitrate

7.3 SAMPLE ANALYSIS

7.3.1 Weigh an aliquot of dried, sieved sample and add to a 500 mL Erlenmeyer flask. Add a stir bar to the flask. The sample aliquot may weigh up to 10 g and should contain 10 to 25 mg of organic carbon.

7.3.2 Using a volumetric pipet, add 10.0 mL of 1N potassium dichromate solution to a 500 mL Erlenmeyer flask.

7.3.3 Using a bottle-top dispenser, carefully add 20 mL of concentrated H_2SO_4 to the flask. Perform this operation in a fume hood. Swirl the flask to mix the reagents and place on a stir plate and stir for one minute to thoroughly mix the sample and reagents. Allow to cool in the fume hood for 30 minutes.

7.3.4 Using a graduated plastic measuring cup, add 200 mL of reagent water to the flask and swirl to mix. If the suspension is very turbid and the titration endpoint will be difficult to discern, quantitatively filter the suspension through a 1.2 uM glass fiber filter using a vacuum filtration apparatus. Rinse the apparatus and filter pad with a small amount of reagent water (10 – 20 mL) to ensure that the filtration is quantitative. The small amount of added water will make no difference in the titration.

7.3.5 Add 5 to 10 drops of ferroin indicator to the flask and place a Teflon stir bar to the flask. Stir the mixture continuously on a stir plate while titrating with the ferrous sulfate titrant until the endpoint is reached. Record the volume of titrant used to the nearest 0.05 mL.

7.3.6 If all of the dichromate ion has been consumed in the sample preparation (i.e. if the sample contains organic carbon in excess of the amount of dichromate added), the sample will turn reddish-brown as soon as the first drops of titrant are added. In this case, or if less than 4.0 mL of titrant are used in the titration, reanalyze the sample using a smaller sample aliquot.

7.3.7 Calculate the amount of organic carbon in the sample according to the following equation:

$$\text{Organic Carbon, Wt. \%} = \frac{[10-(\text{Normality of } FeSO_4 \times \text{mL of } FeSO_4)](0.003)(100)(1.3)}{\text{grams of dry soil}}$$

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 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 Prepare and analyze a Laboratory Control Sample (LCS) in each analytical batch of 20 or fewer samples. The LCS is prepared by adding 1.0 mL of 10000 mg/L TOC standard (Section 5.6) to 1.0 grams of blank sand in a 500 mL Erlenmeyer flask.

The expected TOC concentration of the LCS is 1.0%, Acceptance criteria for the titrimetric LCS is 80-120% recovery If LCS is out-of-criteria, reanalyze prior to beginning the titrations of the samples. If the LCS remains out, check the standardization of the ferrous sulfate titrant. Reprepare the associated reagents and standards and repeat the LCS.

8.2 Prepare and analyze a Method Blank in each analytical batch of 20 or fewer samples. The Method Blank consists of 10 g of blank sand. Acceptance criteria for the method blank is an organic carbon concentration less than or equal to the PQL (0.1% by weight).

8.3 Analyze one sample in duplicate for every 10 or fewer samples. Acceptance criteria for the laboratory duplicate is a relative percent difference of 20% or less if the sample concentration is at least three times the PQL.

8.4 Prepare and analyze one matrix spike sample in each analytical batch of 20 or fewer samples. Matrix spikes are prepared by adding 0.5 mL of 10000 mg/L TOC standard (Section 5.6) to a dry sample aliquot in a 500 mL Erlenmeyer flask prior to sample preparation and analysis. Acceptance criteria for the titrimetric matrix spike are 75-125% recovery. If matrix spike recovery or duplicate RPD is out of criteria narrate or flag appropriately.

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

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

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 376.1 and SW846 Method 9034 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Walkley, A. and I.A. Black. 1934. "An Examination of Degtjareff Method for Determining Soil Organic Matter and a Proposed Modification of the Chromic Acid Titration Method". Soil Science 37:29-37.

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, US EPA SW-846, 3rd Edition, Method 9034, Rev. 0, December, 1996.

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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.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Titrimetric Deter-mination of Organic Carbon (Walkley-Black)	Method blank	One per prep batch of 20 or fewer samples	No analyte detected >PQL	(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 where possible or flag results with "B"
	LCS	One per prep batch of 20 or fewer samples	80% - 120% Recovery	(1) If the LCS fails repeat LCS determination (2) Reprepare affected reagent or standard and repeat LCS
	Matrix Spike	One for every set of 20 or fewer samples	75% - 125% Recovery	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. Low recovery may be due to acid-insoluble organic carbons.
	Sample Duplicate	One sample duplicate per ten samples	(1) RPD $\leq$20 for sample results greater than 3 times the PQl (2) For sample results less than three times the PQL, the RPD between sample and duplicate should be < 100%.	(1) If RPD is outside criteria report original result with notation or narration.
	Demonstration of analyst proficiency – 4 replicates of LCS	Once per analyst per year	P&A meet method criteria	Repeat P&A study
	MDL study and/or LOD/LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-777-00	Walkley Black Method
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

FIGURE 1

EXAMPLE OF SAMPLE PREPARATION LOGBOOK PAGE

SAMPLE PREPARATION FOR TOC IN SOIL (WALKLEY-BLACK TITRATION)

Oven ID:	
Analyst In:	Analyst Out:
Date In:	Date Out:
Time In:	Time Out:
Temp In (°C):	Temp Out (°C):

Sieve Serial No: 12207200

Balance ID:		
True Wt. (g)	Initial Wt. (g)	Final Wt. (g)
1.0000		
10.0000		
50.0000		
100.0000		

Sample ID	Dish ID	Dish Wt. (g)	Wet Sample + Dish Wt. (g)	Wet Sample + Dish Wt. (g)

Sample grinding performed by:	**on:**
Sample sieving performed by:	**on:**
Reviewed by:	**on:**

WL-054 – Revision 1 – 04/29/2013

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

FIGURE 2

EXAMPLE OF SAMPLE ANALYSIS LOGBOOK PAGE

TOC IN SOILS BY WALKLEY-BLACK TITRATION

Reagent IDs

Potassium Dichromate: _____ Ferroin Indicator: _____
Ferrous Sulfate Titrant: _____ H2SO4 Lot No.: _____
TOC Standard (10000 ppm): _____

Standardization of Ferrous Sulfate Titrant

mL $K_2Cr_2O_7$ used: _____
mL Titrant used: _____ Normality = mL $K_2Cr_2O_7$ / mL Titrant
Ferrous Sulfate Normality: _____

Sample Analysis

Sample ID	Weight of Dried Sample Used (g)	mL 10000 ppm TOC Std. Added	mL Ferrous Sulfate Titrant Used	Analysis Time

Analyst: _____ Reviewed By: _____
Analysis Date: _____ Review Date: _____

WL-055 – Revision 1 – 05/15/2013

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

FIGURE 3

EXAMPLE OF CALCULATION SPREADSHEET

TOC in Soil (Walkley-Black Titration) **Analysis Date: 5/16/2013**

FeSO4 Titrant Normality 0.500
Spiking Std. Conc. (mg/L) 10000

Sample	Sample Wt. (g)	Titrant Vol. (mL)	TOC (mg)	TOC (mg/kg)	TOC (Wt %)	Corrected TOC (Wt %)	Spk Std Added (mL)	TOC Spk Added (Wt %)	Dup RPD	Rcvy. (%)
Blank	10.0000	20.2	-0.30	-30.00	-0.0030	-0.0039				
LCS	10.0000	14.1	8.85	885.00	0.0885	0.1151	1.0000	0.1300		88.5
SG2869-6	0.5009	5.5	21.75	43421.84	4.3422	5.6448				
SG2869-6DUP	0.5013	6.0	21.00	41891.08	4.1891	5.4458			3.6	
SG2869-6MS	0.5007	3.3	25.05	50029.96	5.0030	6.5039	0.5000	1.2982		66.2
SG2869-7	0.5002	11.7	12.45	24890.04	2.4890	3.2357				
SG2869-9	0.5015	12.8	10.80	21535.39	2.1535	2.7996				
SG2869-10	0.5012	13.3	10.05	20051.88	2.0052	2.6067				
SG2869-13	0.5018	8.2	17.70	35273.02	3.5273	4.5855				
SG2869-13DUP	0.5012	8.1	17.85	35614.53	3.5615	4.6299			1.1	
SG2869-14	0.5014	13.3	10.05	20043.88	2.0044	2.6057				
SG3026-11	0.5000	17.5	3.75	7500.00	0.7500	0.9750				
SG3026-12	0.5010	18.3	2.55	5089.82	0.5090	0.6617	Rerun with larger sample aliquot			
SG3026-13	0.5006	19.8	0.30	599.28	0.0599	0.0779	Rerun with larger sample aliquot			
SG3026-14	0.5007	16.0	6.00	11983.22	1.1983	1.5578				
SG3026-15	0.5020	19.8	0.30	597.61	0.0598	0.0777	Rerun with larger sample aliquot			
SG2869-8	0.1003	16.0	6.00	59820.54	5.9821	7.7767				
SG2869-11	0.1008	15.5	6.75	66964.29	6.6964	8.7054				
SG2869-12	0.1008	16.8	4.80	47619.05	4.7619	6.1905				
SG2869-15	0.1009	17.1	4.35	43111.99	4.3112	5.6046				

TITLE: TITRIMETRIC DETERMINATION OF ORGANIC CARBON USING THE WALKLEY-BLACK METHOD

FIGURE 4

EXAMPLE OF BATCH SHEET

WET CHEMISTRY BATCH REPORT
May 22 2013, 09:25 am
Batch: WG124184

Parameter: Total Organic Carbon

Date Analyzed: 15-MAY-13

Analyst Initials: KP

Prep Date: 02-MAY-13

Prep Method: EPA 821

Prep Chemist: KP

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SG2869-2	SAMP	WALKLEY-BLAC	0.50100mg			2	2.0 wt. % dry	60.	.1	0.010	0.10		
SG2869-3	SAMP	WALKLEY-BLAC	0.10200mg			9.8	9.8 wt. % dry	34.	.1	0.010	0.10		
SG2869-4	SAMP	WALKLEY-BLAC	0.10300mg			9.3	9.3 wt. % dry	27.	.1	0.010	0.10		
SG2869-5	SAMP	WALKLEY-BLAC	0.11000mg			5.8	5.8 wt. % dry	43.	.1	0.010	0.10		
WG124184-1	MBLANK	WALKLEY-BLAC	10.000mg			0	U0.040 wt. % dry	NA	.1	0.010	0.10		
WG124184-2	LCS	WALKLEY-BLAC	10.000mg			.12	0.12 wt. % dry	NA	.1	0.010	0.10		92
WG124184-3	DUP	WALKLEY-BLAC	0.50100mg			1.8	1.8 wt. % dry	NA	.1	0.010	0.10	10	
WG124184-4	MS	WALKLEY-BLAC	0.50200mg			3.1	3.1 wt. % dry	NA	.1	0.010	0.10		85

Comments:

SG2869-5	MS/MSD
WG124184-1	SG2869-2
WG124184-2	SG2869-2
WG124184-3	SG2869-2
WG124184-4	SG2869-2

Entered by:_____ Date:_____ Accepted by:_____ Date:_____

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Kerri Pawlina

Review Date: 8/29/13

SOP Number: CA777

SOP Title: TOC Walkley Black

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:** 11/18/13

QAO Signature: Susie Dimond **Date:** 11.18.13

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zachary B. Smith

Review Date: 05 - February - 2015

SOP Number: CA - 777

SOP Title: Titrimetric Determination of Organic Carbon using the Walkley - Black Method

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:** 08/20/15

QAO Signature: Leslie Dimond **Date:** 08.20.15

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zach Fuller

Review Date: 2-7-17

SOP Number: CA-777-00

SOP Title: Titrimetric Determination of Organic Carbon using the Walkley-Black Method

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: A. Brewer

Date: 03/08/17

QAO Signature: Lescia Dimond

Date: 03.13.17

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Zach Fuller

Review Date: 2/27/19

SOP Number: CA-777-00

SOP Title: TOC Walkley Black

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: Gary Brewer

Date: 03/15/19

QAO Signature: Leslie Dimond

Date: 04.17.19

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: J. 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.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

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.

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 shirts 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
14	Sect. 7.15.5 - Changed Login COC to Login Details. Updated Figures 2 and 3.	LAD	05/20	05/20

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-902-14**, titled **Sample Receipt and Internal Control**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-14**, 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.

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 Celsius. 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 <u>notes section of the SRCR</u> 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.

<u>Top Section of Screen</u>:

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.

Order Date - 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 Detail”. 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.

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 (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) 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.

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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

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

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

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

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

TABLE 1

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					

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

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

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

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:	

*State Compliance Sample? YES / NO Will KAS report to DWP? YES / NO PWSID #

Lab Use Only | Work Order #:

Cash / Check# Amount#

Sample iced/chilled?_____

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 4 – 10/24/2017

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:
Shipping: UPS Fed-Ex	Airbill No.:	
Temperature:		

Food & Microbiological Services

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobactor	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 2 – 03/05/2019

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 #: HK 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	[illegible]	
5/13/16	1350	5-13-16	15:00	SJ 3312	Bristol Seafood	RJ	
↓	↓			SJ 3313	Camp Sunshine	↓	
5-13-16	14:00			SJ 3314	DEP-B	GW	
				SJ 3315	↓	GW	
				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	~~F~~ PWD	↓	open
↓	↓	5-13-16	16:40	SJ 3335	Cape Elizabeth Tax	GW	
5-13-16	15:00			SJ 3336	MEL	GW	
↓	↓			SJ 3337	↓	↓	
5-13-16	15:35			SJ 3338	PWD	GW	
5/13/16	1540			SJ 3339	Maine Medical	PRS	
↓	↓			SJ 3340	COZM	↓	
↓	↓	↓	↓	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

Holding Time						Immediate			24 Hour				48 Hour										
Work Order	Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Quick TAT Parameters	pH	DO	Sulfite	Fe+2	Cr+6	Total BOD	Carbon BOD	Color	MBAs	Nitrate (Lachat)	Nitrate (IC)	Nitrite (Lachat)	Nitrite (IC)	OPO4 (Konelab)	OPO4 (IC)	Set Solids	Turb	Comments
SI3915	CH2MHill	AQ	6/5/2015	1430	1xRCl [1]	1																	
SI3915	CH2MHill	SL	6/5/2015	15:30	1xRCl [2]	1																	
SI3944	AECOM	AQ	6/8/2015	1000	AQx2(2day)-pH	2																	
SI3944	AECOM	SL	6/8/2015	700	SLx3(1wk)-RCl,Paint,Ts [3]																		
SI3948	Drumlin	AQ	6/8/2015	10:30		7																	
SI3949	Drumlin	AQ	6/8/2015	11:30		1																	
SI3953	Bethel WWTP	SL	6/8/2015	11:00											1		1						
SI3954	CMP	FP	6/5/2015	15:00	ss=%100																		
SI3981	Pineland Farms	AQ	6/9/2015	7:30		1					1										1		
SI3987	Strem	AQ	6/9/2015	10:30		1					1												
SI3988	Strem	AQ	6/9/2015	10:30		1					1												
SI3989	Nimby Env	SL	6/8/2015	8:00											2		2						
SI3993	Acres of Wildlife	AQ	6/9/2015	10:04											4		4						
SI3995	Clean Harbors	AQ	6/10/2015	4:00	COD, Ammonia,F,Flash,CN,BOD [4]						1												8hr TAT
SI3996	HO	AQ	6/9/2015	13:33		1									1		1						
SI4001	Lockheed	AQ	6/9/2015	10:15							4					4		4					
SI4004	Ogunquit SD	SL	6/9/2015	8:30		1									1		1						
SI4006	Homeowner	aq	6/10/2015	10:30											1		1						
SI4009	USDA	aq	6/9/2015	15:00											1		1						
SI4010	CCAR	aq	6/9/2015	1500											1		1						
SI4011	MFTL	aq	6/9/2015	1400							1												
SI4019	allagash	aq	6/10/2015	1113	2x caustic alk						1												
SI4020	kerry	aq	6/10/2015	1139							1												
SI4021	city of saco gen d	aq	6/10/2015	1220						1													
SI4022	strem	aq	6/10/2015	1000		1					1												
SI4023	strem	aq	6/10/2015	1000		1					1												
SI4041	LGS	AQ	6/10/2015	1330		3									3								moved from bod to nitrate
SI4042	LGS	AQ	6/10/2015	1350		2									2								moved from bod to nitrate
SI4047	Clean Harbors	SL	6/10/2015	1100		6																	
					LAST CHECKED 18:31 06-10-15 DW																		

Holding Time						Immediate			24 Hour				48 Hour										
Work Order	Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Quick TAT Parameters	pH	DO	Sulfite	Fe+2	Cr+6	Total BOD	Carbon BOD	Color	MBAs	Nitrate (Lachat)	Nitrate (IC)	Nitrite (Lachat)	Nitrite (IC)	OPO4 (Konelab)	OPO4 (IC)	Set Solids	Turb	Comments
SI4048	Windham	Aq	6/10/2015	1830								2											
SI4051	Ch2mHill	SL	6/10/2015	1045	RClx1 [5]																		1 wkTAT
SI4052	Lockheed	AQ	6/10/2015	940							6					6 [6]		6 [7]					
SI4053	Pineland	AQ	6/10/2015	1300							1				1								
SI4065	ISF	AQ	6/11/2015	900																	1		
SI4067	YCC	AQ	6/10/2015	1330											1		1						
SI4069	Twin Rivers	AQ	6/9/2015	1545									3 [8]										
SI4077	Strem	AQ	6/11/2015	915		1					1												
SI4078	Strem	Aq	6/11/2015	915		1					1												
SI4070	Maine Electronics	AQ	6/10/2015	945		3																	
SI4087	GAUD	SL	6/10/2015	1400		1									1		1						
SI4089	EcoMaine	AQ	6/11/2015	1115	caust alk x 2	2					1												
SI4104	HO	AQ	6/11/2015	1700		1									1		1						
											1												

Holding Time						Immediate			24 Hour				48 Hour										
Work Order	Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Quick TAT Parameters	pH	DO	Sulfite	Fe+2	Cr+6	Total BOD	Carbon BOD	Color	MBAs	Nitrate (Lachat)	Nitrate (IC)	Nitrite (Lachat)	Nitrite (IC)	OPO4 (Konelab)	OPO4 (IC)	Set Solids	Turb	Comments
SI4106	Lockheed	AQ	6/11/2015	1010							4					4		4					MS/MSD
si4112	srcc	aq	6/12/2015	620															1				

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

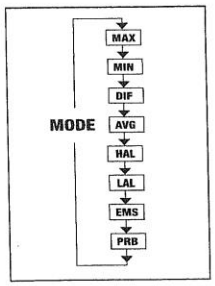
IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

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.

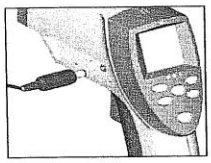
Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

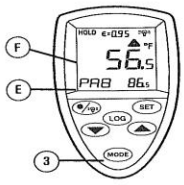
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.



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.





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.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No.	Site ID	Filtration Requested By:		Filtered and Preserved By:		
List Individually	(Optional)	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

Cert No E87604

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
						$ 385.00

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

Page: *1 of 1*

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	:

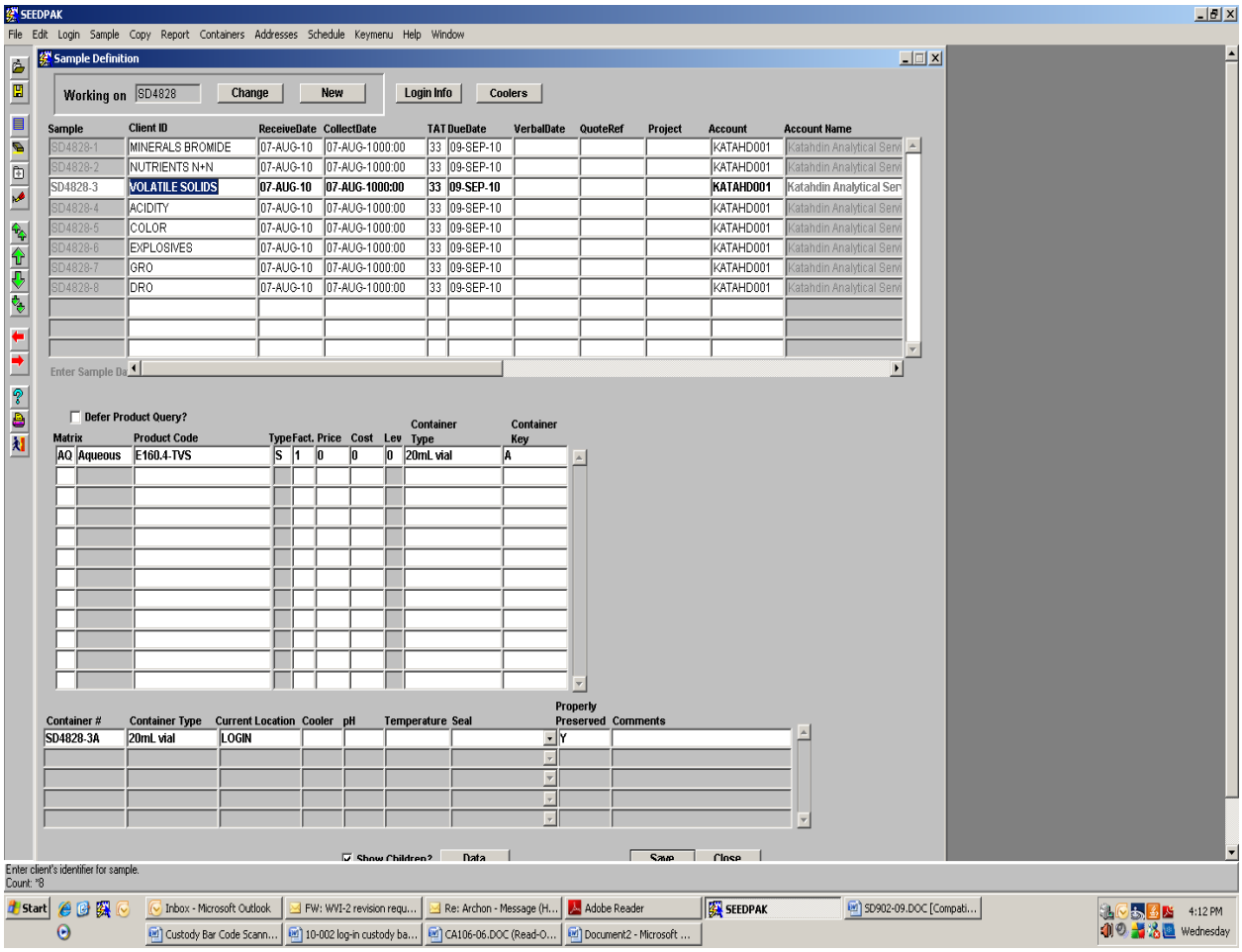
Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	***Product***	***Hold Date (shortest)***	***Bottle Type***		***Bottle Count***		
Aqueous S	SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	***Product***	***Hold Date (shortest)***	***Bottle Type***		***Bottle Count***		
Aqueous S	SW8260FULL5ML	09-FEB-07			2		

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: Joel C. Benton Date: 2/01

QA Officer: Deborah J. Nadeau 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.	LAD	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

Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations
Figure 5 Example of Elementary Neutralization Logbook

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		4-13-13	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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TITLE: SAMPLE DISPOSAL

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):					

HW-007 – Revision 2 – 08/30/2017

0000029

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: GN
# 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: GN/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: Lucie Dimond **Date:** 01.10.20

TITLE: CATION-EXCHANGE CAPACITY (CEC) OF SOILS

SW-846 Method 9081 and LDNR Method 29-B

Approvals (Signature/Date):

John Mathewson 11/03/20 John Mathewson Date Inorganics Department Manager	John Mathewson 11/03/20 John Mathewson Date Environmental Health & Safety Coordinator
Tiffany Fleming 11/03/20 Tiffany Fleming Date Quality Assurance Coordinator	Chip Meador 11/03/20 Chip Meador Date Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) provides procedure used by Eurofins TestAmerica Corpus Christi for determining the Cation Exchange Capacity of soil samples.

1.2 This SOP is based on SW-846 Method 9081 and LDNR Method 29-B.

1.3 The procedure involves the extraction of the solid sample with first a sodium acetate solution, then with an ammonium acetate solution. The displaced sodium is then measured in the ammonium acetate extract using ICP/ICPMS analysis.

1.4 The typical Reporting limit for this method is 0.05meq/100gm.

2.0 SUMMARY OF METHOD

2.1 The soil is mixed with an excess of sodium acetate solution. This results in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaced the adsorbed sodium with ammonium. The concentration of displaced sodium is then measured in the ammonium acetate extract using ICP/ICPMS analysis.

3.0 DEFINITIONS

3.1 Refer to SW-846 Method 9081 for definitions of terms used in this SOP.

3.2 LDNR – Louisiana Department of Natural Resources.

3.3 TALS - TestAmerica Laboratory System.

3.4 NCM – Non-Conformance Memo. generated in the TALS.

4.0 INTERFERENCES

4.1 See SOP CC-ATM-M005/CC-ATM-M025, current revision, for ICP/ICPMS Analysis by SW-846 Method 6010/6020 or equivalent SOP.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1.1 Method Specific Safety: None

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5.1.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Isopropanol alcohol 99%	Flammable	400 ppm-TWA	Flammable liquid and vapor. Harmful if swallowed or inhaled. Causes irritation to eyes and respiratory tract. Affects central nervous system. May be harmful if absorbed through skin. May cause irritation to skin.
Sodium hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or sever burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium acetate	Irritant	None	Do not ingest. Avoid contact with eyes, skin and clothing. Use only with adequate ventilation. Wear appropriate respirator when ventilation is inadequate. Keep in the original container or an approved alternative made from a compatible material, kept tightly closed when not in use. Empty containers retain product residue and can be hazardous. Do not reuse container.
Ammonium acetate	Irritant	None	May cause irritation to skin, eyes, and respiratory tract. May be harmful if swallowed.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Top Loading Balance

6.2 Laboratory Centrifuge

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6.3 Mechanical shaker

6.4 Centrifuge tubes: 50 mL

6.5 Digestion tubes with mL markings

6.6 Graduated cylinders, Class A

6.7 Volumetric flasks, Class A

6.8 pH test strips

7.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests.

Shelf-Life: Reagents received from the vendor expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.1 Ammonium Hydroxide (NH_4OH), concentrated

7.2 Glacial acetic acid

7.3 Ammonium Acetate (NH_4OAc), 1N:

7.3.1 Dilute 57 mL of glacial Acetic Acid (99.5%) with reagent water to a volume of approximately 500 mL. Then add 69 mL concentrated Ammonium Hydroxide, mix, and add reagent water to obtain a volume of about 980 mL. Check the pH of the resulting solution; add more NH_4OH, as needed, to obtain a pH of 7 ± 0.05, and dilute the solution to a volume of 1 liter with reagent water. Record the pH in the batch information sheet in TALS.

7.3.2 Bring to the 1 liter mark with DI water. Shake well.

7.4 Sodium Acetate, 1N:

7.4.1 Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ (sodium acetate) in about 500 mL reagent water and dilute to 1 liter final volume. The pH of this solution should be 8.2 ± 0.05. If needed, add a few drops of Acetic acid or Sodium Hydroxide (10N) solution to bring the solution to pH 8.2. Record the pH in the batch information sheet in TALS. Check the pH; adjust to 8.2 with acetic acid or NaOH, if needed.

7.4.2 Bring to the 1 liter mark with DI water. Shake well.

7.5 Isopropyl alcohol: 2-propanol 99%

7.6 DI water: Deionized, type I reagent water

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

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8.1 Sample Collection, Preservation, Storage and Holding Times: Soil samples are collected in plastic or glass 1000 mL sample containers, and stored at 4°C. The holding time for extraction and analysis is 6 months from date of collection.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	HDPE or Glass	20 grams	No requirement	6 months	SW-846 Chapter 2

Temperature preservation is not used.

9.0 QUALITY CONTROL

9.1 Refer to the individual analytical method used for determining sodium for quality controls to be followed.

9.2 Documentation of support equipment and lot numbers of all consumable items will be recorded for each prep batch in TALS.

9.3 Demonstration of Proficiency: Each laboratory analyst must complete initial and ongoing demonstration of capability for each method used. Refer to employee training records.

9.4 Method Detection Limits: Not applicable to this method; refer to method used for determining sodium.

9.5 Batch: A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.6 Instrument QC: Refer to SOPs CC-ATM-M005 for ICP and CC-ATM-M025 for ICP-MS, current revisions.

9.6 Sample QC: A material of known cation-exchange capacity will be routinely analyzed.

9.7 Sample QC: The following quality control samples are prepared in each batch:

9.7.1 Method Blank (MB): The blank is de-ionized water taken through the procedure as if it were a sample. A method blank is required with every batch of 20 or less samples. The MB must be less than the Reporting Limit (RL).

9.7.2 Sample Duplicate: One sample duplicate is required at a 10% frequency (one per 10 samples or less).

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per batch	< RL	Correct problem, then re-prep and reanalyze all samples and QC in the affected analytical batch.
Sample Duplicate	One per batch	<20% RPD	Report.
QC sample of a known CEC	Routinely analyzed	>RL	Correct problem, then re-prep and reanalyze all samples and QC in the affected analytical batch.

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10.0 PROCEDURE

10.1 Weigh out and label samples

10.1.1 Evaluate sample grain size and document the review for each sample in the TALS prep batch.

10.1.2 For RCRA Cation Exchange Capacity, weigh 4 +/- 1 grams of medium- or fine-textured soil or 6 +/- 1 grams of coarse-textured soil and transfer the sample to a 50-mL centrifuge tube. (A fine soil has > 50% of the particles ≤ 0.074 mm, medium soil has >50% ≥ 0.425 mm, while a coarse soil has more than 50% of its particles ≥ 2 mm.) Record the soil weight used.

10.1.3 For LDNR 29-B Cation Exchange Capacity, prepare 5.0 ± 1 grams as described in SOP CC-ATM-WC063, current revision, for LDNR 29-B Soil Preparation.

10.1.4 Label one centrifuge as “prep blank” or “method blank”. This procedure requires one blank per 20 samples and one sample duplicate per 10 samples.

10.2 Sample preparation:

10.2.1 Sodium Acetate

10.2.1.1 Add 33mL of 1N sodium acetate to each centrifuge tube. Cap the tubes. Place on a shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes at 2000 RPM or until the supernatant liquid is clear. Decant the liquid. (Do not save).

10.2.1.2 Repeat step 10.2.1.1 **three more times**.

10.2.2 Isopropyl alcohol (2-propanol)

10.2.2.1 Add 33 mL of 70-90% isopropyl alcohol to each centrifuge tube. Cap the tubes. Place on shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes at 2000 RPM or until the supernatant liquid is clear. Decant the liquid. (Do not save).

10.2.2.2 Repeat step 10.2.2.1 **two more times**.

10.2.3 Ammonium acetate

10.2.3.1 Add 33 mL ammonium acetate to each centrifuge tube. Cap the tubes. Place on shaker and shake for 5 minutes. Place the tubes in the centrifuge and spin for about 15 minutes at 2000 RPM or until the supernatant liquid is clear. **Decant the 1 N ammonium acetate extract into a 100-mL volumetric flask.**

10.2.3.2 Repeat step 10.2.3.1 **two more times**.

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10.2.4 Dilute the combined washing (1N ammonium acetate) to the 100-mL mark with 1N ammonium acetate solution.

10.3 Sample Analysis:

10.3.1 Analyze the batch for sodium (Na) using ICP Method 6010 or ICP-MS Method 6020.

11.0 DATA GENERATION AND CALCULATIONS

11.1 Data Generation

The data to be generated and recorded are the measured concentration values of sodium in units of mg/L.

11.2 Calculation of CEC

$$\frac{\text{Na mg/L}}{23} = \text{meq/L (in the extract)}$$

$$\text{CEC} = \frac{\text{Meq Na x 10}}{\text{Sample wt in gm (4 g)}} = \text{meq / 100g soil}$$

11.3 Calculation of Relative Percent Difference for Duplicates

$$\text{RPD} = \frac{|A - B|}{\frac{1}{2}(A + B)} \times 100\% = \frac{|A - B|}{(A + B)} \times 200\%$$

Where: A = concentration of analyte in original sample
B = concentration of analyte in duplicate sample

12.0 DATA ASSESSMENT AND CORRECTIVE ACTION

12.1 Data Assessment

12.1.1 The analyst does primary data review at the time of analysis.

12.1.2 The section supervisor or senior analyst does secondary data review.

12.1.3 The section supervisor, project manager, QA Manager, or Laboratory Director does completeness review and approval.

12.2 Handling Out-of-Control or Unacceptable Data and Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with

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the out-of-control situation should be reanalyzed. Out-of-control data must not be released without approval of the supervisor or QA personnel.

13.0 DATA REDUCTION AND REPORTING

13.1 Data Reduction

The analytical value is entered into TALS manually and the analytical value is converted to the reported value by the application of the calculation in Section 11.2.

13.2 Raw Data Entry and Reporting

13.2.1 Report the results of the calculation as “CEC” in units of meq/ 100gm (milliequivalents per 100 grams). All information pertaining to sample preparation and analysis must be entered as it is acquired on the computer generated run logs, or instrument printout, or designated bound logbook. This includes the test method: analyte(s); matrix; full ID for samples, analyst’s initials; and date/time of calculations. All information and data pertinent to the method and/or analysis should be recorded to facilitate data validation. Any information that is important or may influence the test results should be noted, such as sample appearance, problems encountered, unusual conditions, reasons for repeated tests or rejected, etc.

13.2.2 Instrument printouts must be initialed and dated by the instrument operator.

13.2.3 Instrument generated data must be uniquely identified and maintained according to the laboratory’s procedure for data storage.

13.2.4 Any data that does not meet the method/project requirements or QC specifications must be documented on a NCM. Document the out of control event or nonconformance, the sample(s) and parameter(s) affected, and the corrective action taken. The NCM is used to generate a case narrative to include with the final data report.

14.0 METHOD PERFORMANCE:

14.1 Refer to method used for performance data.

14.2 Section supervisor/group leader has the responsibility to ensure that this procedure is performed by an associate who has been trained in its use and has the required experience.

15.0 POLLUTION CONTROL

15.1 It is Eurofins TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

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16.0 WASTE MANAGEMENT

16.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

16.2 Waste Stream Produced by the Method: All samples and standards are placed into the laboratory acid waste stream for disposal.

17.0 REFERENCES / CROSS-REFERENCES

17.1 Method 9081, Test Method for Evaluating Solid Waste, USEPA SW-846, 1986.

17.2 Methods 29-B, Laboratory Procedures for Analysis of Exploration & Production Waste, Louisiana Department of Natural Resources, Revision 4, May 2005.

17.3 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision.

17.4 Eurofins TestAmerica Quality Assurance Manual (CC-QAM), current revision.

17.5 Associated SOPs:

17.5.1 CC-ATM-M005, Trace Metals by ICP-AES (6010B and 200.7), current revision.

17.5.2 CC-ATM-M025, Trace Metals by ICP-MS (6020 and 200.8), current revision.

17.5.3 CC-ATM-WC034, Soil and Waste pH, current revision.

17.5.4 CC-ATM-WC063, Soil Preparation (LDNR 29-B), current revision.

17.5.5 CC-GLO-001, Documentation Procedures, current revision.

17.5.6 CC-GLO-002, General Glassware Cleaning, current revision.

17.5.7 CC-GLO-014, Maintenance and Calibration of Balances and Class I Weights, current revision.

18.0 METHOD MODIFICATIONS:

18.1 To minimize waste generated, the Eurofins TestAmerica Corpus Christi laboratory prepares samples at half the weights/volumes specified in Section 10.0.

19.0 ATTACHMENTS, TABLES, ASSOCIATED DOCUMENTS AND FORMS

19.1 Table 1: Equivalent weights table for major Cations and Anions

19.2 Table 2: Conversion factors for mg/L to meq/L

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20.0 REVISION HISTORY

Historical File:		
	Revision 00: 05/25/04	Revision 01: 08/07/06
	Revision 02: 02/26/08	Revision 03: 04/26/09
	Revision 04: 06/10/10	Revision 05: 05/27/15
	Revision 06: 06/07/17	Revision 07: 01/31/19
	Revision 08: 05/20/19	Revision 09: 11/03/20

20.1 Revision 09, dated 03 November 2020, updated by Tiffany Fleming

20.1.1 Updated section 7 to include documentation of pH results;

20.1.2 Updated section 9 to include documentation of support equipment and a reference material;

20.1.3 Updated section 10.1 with additional detail;

20.1.4 Updated section 10.2 to include centrifuge speed

20.2 Revision 08, dated 20 May 2019, updated by Christina Godines:

20.2.1 Updated with rebranding throughout;

20.2.2 Added reference to LDNR 29-B in Sections 1.2, 7.0, 17.0;

20.2.3 Added LDNR to Definitions in Section 3.2;

20.2.4 Updated Sections 7.3 and 7.4 with additional detail;

20.2.5 Added LDNR detail and reformatted Section 10.0;

20.2.6 Updated References to include LDNR and Cross-references to include new soil prep SOP in Section 18.0;

20.2.7 Added method modification in Section 19.0 to use half volumes/weights during sample preparation.

20.3 Revision 07, dated 31 January 2019, updated by Christina Godines:

20.3.1 Removed ‘Sample Disposal’ from Section 5.0;

20.3.2 Updated wording in Section 6.0 and removed ‘Waste Management and Pollution Prevention’ (added as Sections 15.0 and 16.0);

20.3.3 Renamed Section 8.0 to ‘Equipment and Supplies;’

20.3.4 Added ‘Cross-references’ to Section 14.0 name and added related SOPs;

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20.3.5 Moved Section 15.0 to Section 14.0; renamed15.0 to Method Modifications;

20.3.6 Moved Section 5.0 to Section 8.0;

20.3.7 Moved Section 7.0 to Section 9.0, added QC parameters and detail including Batch, Method Blanks, and Sample Duplicate;

20.3.8 Added Calculation of RPD for duplicates in Section 11.0;

20.3.9 Biennial review and update.

20.4 Reason for changes, Revision 05:

20.4.1 Updated Section 10.4.1 to reflect current use of 70-90% isopropyl alcohol.

Table 1: Equivalent weights table for major Cations and Anions

Equivalent Weights Table for the Major Cations and Anions			
Cations		**Anions**	
Element	Equivalent Weight	Element	Equivalent Weight
Calcium (Ca)	20.04	Chloride (Cl)	35.453
Magnesium (Mg)	12.1525	Carbonate (CO3)	30.004
Sodium (Na)	22.9898	Bicarbonate (HCO3)	61.016
Potassium (K)	39.098	Sulfate (SO4)	48.03
Lithium (Li)	6.94	Nitrate (NO3)	62.004
Iron (Fe)	27.97	Bromide (Br)	79.904
Manganese (Mn)	27.469	Nitrogen (N)	14.007
Strontium (Sr)	43.81	Ammonium (NH4)	18.039
Barium (Ba)	68.665	Phosphate (PO4)	31.66

Table 2: Conversion factors for mg/L to meq/L

Contituent	To convert mg/L to meq/L multiply by	To convert meq/L to mg/L multiply by
Calcium (Ca)	0.0499	20.04
Magnesium (Mg)	0.0823	12.15
Sodium (Na)	0.0435	22.99
Potassium (K)	0.0256	39.098
Aluminum(Al)	0.1112	8.99
Manganese (Mn)	0.0364	27.47
Barium (Ba)	0.0146	68.665
Chromium (Cr)	0.0577	17.33
Copper (Cu)	0.0315	31.75
Hydrogen (H)	0.9921	1.008

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NH4	0.0554	18.04
Br	0.0125	79.9
Cl	0.0282	35.45
F	0.0526	18.99
Bicarbonate (HCO3)	0.016	61.016
Carbonate (CO3)	0.033	30.004

TITLE: TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES)

EPA SW-846 METHODS 6010B and 200.7

Approvals (Signature/Date):

John Mathewson 11/20/20 John Mathewson Date Inorganic Department Manager	John Mathewson 11/20/20 John Mathewson Date Environmental Health & Safety Coordinator
Tiffany Fleming 10/13/20 Tiffany Fleming Date Quality Assurance Coordinator	Chip Meador 10/16/20 Chip Meador Date Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) provides guidance for the analysis of trace metals by inductively coupled plasma atomic emission spectrometry (ICP-AES).

1.2 This SOP is based on EPA SW-846 Method 6010B and EPA Method 200.7.

1.3 Applicable matrices for Method 6010B include water, waste, soil, sludge, sediment, and TCLP extracts. Applicable matrices for Method 200.7 include water and waste.

1.4 The detection limits, sensitivity, and optimum operating ranges for each metal will vary with the sample matrix and ICP instrument model. The typical reporting limits for this method are listed in table 1.

2.0 SUMMARY

2.1 The laboratory uses simultaneous ICP-AES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photo-multiplier tubes or a charge injection device (CID). The photo-currents from the photo-multiplier tubes or a charge injection device (CID) are processed and controlled by a computer system.

2.3 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.0 DEFINITIONS

3.1 Dual View ICP – an ICP equipped with both radial and axial viewing capabilities.

3.2 Dissolved Metals - Those elements which pass through a 0.45-μm membrane. (The sample is acidified after filtration).

3.3 Suspended Metals - Those elements which are retained by a 0.45-μm membrane.

3.4 Total Metals - The concentration determined on an unfiltered sample following vigorous digestion.

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3.5 Refer to EPA Methods 200.7 and 6010B of SW-846 for definitions of terms used in this SOP.

3.6 TALS – TestAmerica Laboratory System

3.7 NCM – NonConformance Memo, generated in the TALS

4.0 INTERFERENCES

4.1 Interferences: Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1 A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result. Refer to Section 3.0 of EPA Method 6010B for additional information on interferences.

4.1.2 Spectral Interferences: Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte signal. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.1.2.1 When running a single-element interferent standard at the upper concentration range, the absolute value of any result for non-spiked elements that exceeds two times the reporting limit requires investigation and corrective action.

4.1.3 Physical Interference: An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation

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and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable, then dilution of the sample may be necessary to overcome the interferences. Where the use of an internal standard might actually degrade the accuracy of the analytical result, sample results may be reported without IS correction.

4.1.4 Chemical Interferences: Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.1.5 Memory Interference: Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. Flushing the system with a rinse blank between samples can minimize these effects. The rinse times necessary for a particular element must be estimated prior to analysis. Until the required rinse time is established, the method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

4.1.6 High Salt Concentrations: High salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 80-120% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measureable analyte, over correction could go undetected if a negative value is reported as zero.

4.2 Comments and Helpful Hints: Routine instrument maintenance and good glassware cleaning procedures can prevent many operational problems and interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Method-Specific Safety

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5.2.1 All standards and samples contain nitric acid. Lab coats, gloves and safety glasses should be worn while handling standards and samples.

5.2.2 The instrument uses an inductively coupled plasma atomic emission spectrometer. The plasma generates a very bright and intense light that is harmful to the eyes. The instrument is equipped with a protective glass to contain this light and should be in place during instrument operation.

5.2.3 The ICP plasma emits strong UV rays and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3 Primary Material Used

The following is a list of the materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Instrumentation

6.1.1 The instrument used for analysis is an inductively coupled plasma atomic emission spectrometer that is equipped with background correction and operated by a

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dedicated workstation under the control of appropriate software. The software should provide for background compensation, system diagnostics, data storage, and calculations. Thermo Fischer ICP 6500E Trace Analyzer IS currently used. Instruments with demonstrated equivalent performance can also be used.

6.1.2 In addition to the ICP, a radio frequency generator and argon gas supply (welding grade or better) are required.

6.2 Laboratory Materials

6.2.1 Adjustable automatic pipettes

6.2.2 Volumetric flasks, 100mL

6.2.3 Bottles, for storing sample digestates, 150mL

7.0 REAGENTS AND STANDARDS

7.1 Shelf-Life: Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.1.1 The expiration date of intermediate concentration standards or working standards cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.1.2 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately

7.1.3 Standards used for calibration and quality control purposes must be NIST traceable, where available.

7.1.4 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the TALS database.

7.1.5 Calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 9.16 of this procedure.

7.2 Reagents

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7.2.1 DI water

7.2.2 Hydrochloric acid, concentrated

- Life of reagent: 1 year from receipt date
- Storage requirements: store at room temperature

7.2.3 Hydrochloric acid, 1:1 in reagent water

- Life of reagent: six months
- Storage requirements: store at room temperature

7.2.4 Nitric acid, concentrated

- Life of reagent: manufacturer's expiration date
- Storage requirements: store at room temperature

7.2.5 Nitric acid, 1:1 in reagent water

- Life of reagent: six months
- Storage requirements: store at room temperature

7.3 Standards

7.3.1 Stock Standard Solutions: Standard stock solutions are purchased from various vendors. Follow manufacturer's expiration date and storage requirements.

7.3.2 Mixed Calibration Standard Solutions: Prepare calibration standards at the same acid concentration as the calibration blank. Calibration standards are standard solutions containing mixtures of compatible elements that may be purchased and used for calibration. The calibration standard must be initially verified using a quality control sample and monitored weekly for stability. Prepare fresh calibration standards as needed.

7.3.3 Continuing Calibration Verification Standard: The continuing calibration verification (CCV) should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve.

7.3.4 Initial Calibration Verification Standard: The initial calibration verification (ICV) stock standards are purchased from a different vendor than is used to supply the initial calibration standards (ICAL). The ICV is analyzed immediately after the ICAL.

7.3.5 Interference Check Solution (ICSA/ICSAB): The interference check solution is obtained commercially from an outside source.

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8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Collection, Preservation, Storage and Holding Times: Refer to Chapter 3 and Table 3-2 of EPA SW-846 revision 4, February 2007 for additional information on sample collection preservation, storage and holding times. Eurofins Xenco Corpus Christi supplies sample containers and chemical preservatives in accordance with the method. Eurofins Xenco Corpus Christi does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures.

	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	500 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

The exception is the analysis of dissolved silica by Method 200.7, which must be analyzed within 28 days from the date of collection.

Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.1.1 The samples are to be collected in 1-L plastic or glass containers that have been cleaned according to EPA specifications. A minimum volume of 500 mL should be collected for aqueous samples. Preserve aqueous samples with nitric acid to a **pH of <2** with the exception of samples that are to be extracted (i.e. EP Toxicity or TCLP). After extraction, the EP Toxicity or TCLP sample extracts are preserved with nitric to a pH of <2. The maximum holding time for preserved samples is 6 months from the date of sampling.

8.1.2 **Dissolved metals**: If samples are to be analyzed for dissolved metals, the samples must be field filtered within 15 minutes of collection and before adding preservatives. Rinse and condition the filter with 50 to 100mL of sample, discard this portion of filtrate, and then collect 500mL of sample. Acidify the collected sample filtrate with HNO_3 to a pH of ≤ 2.

8.1.3 If the samples are to be analyzed for dissolved metals and are not field filtered, the sample must be filtered through a 0.45um filter as soon as possible upon laboratory receipt. The filtered sample will then be preserved to a pH < 2 with HNO_3. The 24 hour wait period after addition of acid is not necessary in this case (for sample received unfiltered). However, an NCM must be filled out noting that the sample was not filtered at the field.

8.1.3.1 Filtration for dissolved metals may be done using a membrane filter of a different pore size as defined in project-specific requirements.

8.1.4 **Total metals**: An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, the acid must have been added immediately within 15 minutes of collection. If for some reason such as high alkalinity the sample pH is verified to be > 2,

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more acid must be added and the sample held for another 24 hours until verified to be pH < 2. The pH of all aqueous samples **must** be tested prior to digestion to ensure that the sample has been properly preserved. For samples received unpreserved, an NCM must be filled out if the samples are analyzed without the 24 hour hold after adding acid.

9.0 QUALITY CONTROL

All quality control measures described in EPA Method 200.7 Section 9.0, EPA Method 6010B Section 8.0, and Chapter One of SW-846 should be followed.

9.1 Demonstration of Proficiency

9.1.1 Initial Demonstration of Capability (IDOC)

Each laboratory analyst must demonstrate initial demonstration of capability (with each sample preparation and analytical method combination it utilizes) by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. Four replicate aliquots of the reference sample (also referred to as a spiked blank or a laboratory control sample (LCS) containing each target analyte must be initially analyzed by the analyst in order to demonstrate the ability to generate acceptable accuracy and precision data.

9.1.2 Continuing Demonstration of Proficiency (DOC or CDOC)

Annual DOC is also required. Analysts must pass four LCSs, QC study, or proficiency (PT) study and document it yearly. LCS samples from actual test runs or results of a PT or QC sample(s) can be used for this purpose.

9.2 Method Detection Limits: Method detection limits (MDLs) shall be initially determined for all compounds of interest for each method using a clean matrix appropriate to the test method, such as laboratory pure water.

9.2.1 Prepare seven standards at three to five times the estimated MDL concentration.

9.2.2 The MDL standards are processed through the entire analytical process, including the digestion.

9.2.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte (99% confidence level). Then, calculate the MDL for each analyte.

9.3 Detectability Check Sample/ MDL Verification (DCS/MDLV): A Detectability Check Sample (DCS) is a reagent matrix spiked with a known amount of analytes at approximately two to three times the calculated MDL. This sample used to check the MDL for reasonableness to verify the lab's ability to detect analytes at the MDL used for calculating the PQL/RL (practical quantitation limit/reporting limit). **This sample is prepared at client/agency/program request as needed**. [The DCS may also be known as the MDL verification Sample]. The DCS can be prepared from either Vendor/Source.

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9.3.1 The DCS should be taken through all the preparatory and determinative steps to verify that a response is detected. This sample should be analyzed quarterly during the method use and randomly rotated through the instruments so that no instrument is excluded.

Acceptance Criteria: The calculated MDL is verified if the MDLV/DCS standard is detected and the result is significantly different than the blank.

Corrective Actions: If the first MDLV/DCS is not detected, the MDLV/DCS standard will be re-prepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

9.4 Linear Dynamic Range (LDR): The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges that may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

9.4.1 LDR Frequency: LDR should be determined every 6 months. By using the high level standard from each ICAL this frequency is met.

9.4.2 LDR Criteria: Recovery within 10% of the true concentration.

9.5 Batch

9.5.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.5.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.5.3 For this analysis, batch QC consists of a Method Blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.6 Initial and Continuing Calibration Blanks (ICB/CCB)

9.6.1 The calibration blank is an aliquot of reagent water prepared with the same type and concentration of acids as the calibration standard(s). Analyze an initial calibration blank (ICB) after the initial calibration verification standard. Analyze the continuing calibration blanks (CCB) after every continuing calibration standard.

Acceptance Criteria: The absolute value of the ICB/CCB result must be < RL or less than 1/10 the concentration found in the associated samples.

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Corrective Action: If the ICB/CCB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

9.6.2 Fill a 4-liter carboy with about 3.5 liters of reagent water. Slowly add the appropriate amount of concentrated HNO_3 and concentrated HCl. Mix carefully.

9.7 Initial Calibration Verification Standard (ICV): The initial calibration verification standard (ICV) is prepared by the analyst 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. Check calibration with an ICV following the initial calibration.

9.7.1 For Method 200.7, the results of the ICV must be within 95-105% of the known value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.7.2 For Method 6010B, the results of the ICV must be within 90-110% of the known value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.8 Continuing Calibration Verification Standard (CCV): The CCV should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve. The CCV is analyzed, after every 10 samples, and at the end of an analytical run. At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve.

9.8.1 The result obtained for each CCV must be within 90-110% of the known value to be in control. If the CCV exceeds these limits, take corrective action by terminating the analysis, correcting the problem, or recalibrating the instrument, and reanalyzing the previous 10 analytical runs.

NOTE: The frequency of the CCV applies to all sample runs.

9.9 Method Blank (MB): The blank is de-ionized water taken through the procedure as if it were a sample. A method blank is required with every batch of 20 or less samples. In Method 200.7, this is referred to as a Laboratory Reagent Blank (LRB).

Acceptance Criteria: The method blank must not contain any analytes of interest above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated

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samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.10 Laboratory Control Sample (LCS): A laboratory control sample (LCS) consists of either a control matrix spiked with analytes which is carried through the sample preparation and analysis procedure as if it were a sample. One LCS is required with each analytical batch. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. The LCS shall be used to determine batch acceptance. In Method 200.7, this is called a Laboratory Fortified Blank (LFB).

Acceptance Criteria: For Method 200.7, the recovery of the LCS must be within 85-115% or tighter.

For Method 6010B, the recovery of the LCS must be within 80-120% or tighter.

In the instance where the LCS recovery is greater than the upper limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

9.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair is required at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/SDS. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. In Method 200.7, this is referred to as a Laboratory Fortified Matrix (LFM).

9.11.1 Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked.

9.11.2 Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The recoveries for the MS and MSD should be within 75-125%. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated).

Corrective Action: If MSD/MSD recoveries fall outside of the established limits and the LCS is in control, the data will be flagged as outside of control limits. Document the results, which are then used by the lab PM to prepare the case narrative to warn the client that the sample result is suspect.

Acceptance Criteria: The relative percent difference (RPD) between the MS and MSD must be within 20%.

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Corrective Action: If the RPD fails to meet precision limit and the recoveries pass, the control limits should be checked as this would be a very rare occurrence if the limits are set properly. If the LCS is in control, it indicates long-term precision, and precision failures within the batch may be due to sample non-homogeneity. MS/MSD results which fall established control limits must be addressed in the narrative. Document the result, which is then used by the lab PM to prepare the case narrative.

9.12 Serial Dilution Test: A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. The test is performed by running a sample at a 5x dilution. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1 to 5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination.

Corrective Action: If the two results do not agree within ± 10%, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and an NCM prepared, which is then used by the lab PM to prepare the case narrative to warn the client the sample result is suspect.

9.13 Post Digestion Spike (PDS):

9.13.1 A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process. In Method 200.7, this is called an “analyte addition test.”

9.13.2 The post digestion spike (PDS) determines any potential matrix interferences. The same sample that was used for the serial dilution test should be used for the PDS.

9.13.3 At Corpus Christi laboratory, matrix interference is determined by evaluating data for the LCS and MS/MSD. Therefore, the PDS is not analyzed unless a client project or program requires it.

9.13.4 Recovery for the PDS should be within 75-125% of the expected results. There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with a NCM.

9.14 Method of Standard Additions (MSA): This technique involves constructing a calibration curve in the sample matrix itself to compensate for a sample interferent that may enhance or depress 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.

9.14.1 Attachment 1 provides more guidance on performing MSA analyses.

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9.15 Interference Check Analysis (ICSA/ICSAB): The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run or at the beginning and end of the run depend on project requirements. In Method 200.7, this is referred to as the Spectral Interference Check (SIC).

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements with reporting limits ≤ 10 µg/L must be ≤ 2 x RL. The absolute value of ICSA results for the non-interfering elements with RLs > 10 µg/L must be ≤ RL.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2 x RL, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2.x RL, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

9.16 Monitoring Internal Standard Results: Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

Acceptance Criteria: For Method 200.7, if the internal standard counts fall within ±30% (70-130%) of the counts observed in the ICAL blank (STD1-Blank), then the data are acceptable.

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For Method 6010B, if the internal standard counts fall within ±50% (50-150%) of the counts observed in the ICAL blank (STD1-Blank), then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the following apply:

The field samples must be diluted and reanalyzed;

The IS concentrations must be raised; or

A different internal standard must be used.

9.17 Quality Control Sample (QCS)

The Method 200.7 requirement for a quarterly QCS is satisfied by the analysis of the second-source ICV, which is analyzed with each initial calibration.

9.18 Background Correction Point: To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength of interest and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations.

9.18.1 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.19 Interelement Corrections (IECs): ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

9.19.1 When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

9.19.2 Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. To determine IECs, run a single element standard at the

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established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

9.19.3 Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace and CID detector instruments as reflected by the ICSA response.

9.20 Procedural Variations/ Nonconformance and Corrective Action

9.20.1 Any variation shall be completely documented using a NCM and approved by the Supervisor or QA Manager.

9.20.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor or QA Manager.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration Standards (ICAL): Instrument calibration is performed daily or every 24 hours using a minimum of three standards and a calibration blank. The concentrations are:

Calibration Standards					
Element	ICBLK mg/L	IC1 mg/L	IC2 / CCV mg/L	IC3 mg/L	IC4 mg/L
Ag	0	0.0025	1	2	
Al	0	0.025	10	20	100
As	0	0.005	1	2	
B	0	0.0025	1	2	
Ba	0	0.0025	1	2	
Be	0	0.0025	1	2	
Bi	0	0.005	1	2	
Ca	0	0.1	40	80	
Cd	0	0.0025	1	2	
Co	0	0.0025	1	2	
Cr	0	0.0025	1	2	

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Cu	0	0.0025	1	2	
Fe	0	0.025	10	20	100
K	0	0.25	100	200	
Li	0	0.005	2	4	
Mg	0	0.1	40	80	
Mn	0	0.025	10	20	
Mo	0	0.0025	1	2	
Na	0	0.025	10	20	500
Ni	0	0.0025	1	2	
P	0	0.005	2	4	
Pb	0	0.005	1	2	
Sb	0	0.05	1	2	
Se	0	0.005	1	2	
Si	0		10	20	
Sn	0	0.005	2	4	
Sr	0	0.0025	1	2	
Ti	0	0.0025	1	2	
Tl	0	0.005	1	2	
V	0	0.0025	1	2	
Zn	0	0.0025	1	2	

10.2 The Low Cal Standard check Solution (CRI): The low level standard is analyzed as required by the programs or projects to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit (RL). The determined concentration should be within ± 30% of the true concentration.

10.3 Initial Calibration Verification/Initial Calibration Blank (ICV/ICB): The initial calibration accuracy is verified by analyzing a second source standard (ICV).

10.3.1 ICV Frequency:

10.3.1.1 Perform with each initial calibration

10.3.2 ICV Criteria:

10.3.2.1 The ICV must fall within 5% (Method 200.7) or 10% (Method 6010) of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures.

Elements	ICV Concs mg/L
Ag	0.5
Al	5
As	0.5
B	0.5
Ba	0.5
Be	0.5
Bi	1

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Ca	50
Cd	0.5
Co	0.5
Cr	0.5
Cu	0.5
Fe	50
K	50
Li	0.5
Mg	50
Mn	5
Mo	0.5
Na	50
Ni	0.5
P	5
Pb	0.5
Sb	0.5
Se	0.5
Si	20
Sn	0.5
Sr	0.5
Ti	0.5
Tl	0.20
V	0.5
Zn	0.5

10.3.3 ICB Frequency:

- An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

10.3.4 ICB Criteria:

10.3.4.1 The ICB result must fall within +/- the RL from zero.

10.3.5 If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, or the instrument recalibrated and the calibration re-verified.

- Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)

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10.4.1 Calibration is monitored throughout the analytical run through the analysis of a known standard.

10.4.2 A CCV may be a second source or the same source as the calibration

10.4.3 CCV Frequency:

- Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

10.4.4 CCV Criteria:

- The CCV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures.

- If a CCV has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then two CCV may be analyzed without any adjustments to the instrument. If the CCV meets criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (ie initial calibration). Samples after the last acceptable CCV require re-analysis.

10.4.5 CCB Frequency:

- A CCB is analyzed with each CCV.

10.4.6 CCB Criteria:

- The CCB result must fall within +/- RL from zero.

10.5 Interference Check Analysis (ICSA/ICSAB)

10.5.1 The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions.

10.5.2 ICSA:

- The ICSA contains only interfering elements.

- Custom multi-element ICS solutions must be used.

- Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

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10.5.3 ICSAB:

- The ICSAB contains analytes and interferents.
- Refer to Table II for the details of ICSAB composition.
- Custom multi-element ICS solutions must be used.

10.5.4 ICSA/ICSAB Frequency:

The ICSA and ICSAB must run with each initial calibration or every 12 hours whichever is shorter.

10.5.5 ICSA/ICSAB Criteria:

- The ICSAB results for interferents must fall within 80 – 120% of the true value.
- ICSA results for the non-interfering elements must fall within ± 2x RL from zero.

ICSA & ICSAB Standard	Elements	Concentration mg/L
ICSA Standard	Fe Al, Ca, Mg	100 250
ICSAB Standard	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	0.25 0.50

- Life of standard: manufacturer's expiration date
- Storage requirements: store at room temperature

10.6 Linear Dynamic Range Verification (LDR): The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges that may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

The linear range is determined for each element on the standard list.

10.6.1 LDR Frequency:

- Methods 200.7 and 6010B stipulate LDR check every 6 months. By using the high level standard from each ICAL this frequency is met.

10.6.2 LDR Criteria:

- Recovery within 10% of the true concentration.

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10.7 Calibration Sequence

- Instrument warm up
- Profile standard
- Initial calibration (3 standards and a blank)
- ICV
- ICB
- CRI (analyzed as required by programs/projects)
- ICSA*
- ICSB*
- CCV
- CCB
- 10 samples
- CCV
- CCB
- 10 samples
- CCV
- CCB

*If sequence is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.

11.0 ANALYTICAL PROCEDURE

11.1 Sample Preparation: Prepare the samples according to the appropriate sample preparation procedure (e.g., EPA SW-846 Methods 3010-3050, 200.7 digestion procedure). Groundwater samples that have been pre-filtered and acidified will not need acid digestion. Refer to sample preparation SOPs for the digestion procedures.

11.2 Instrument Operation, Daily Checks, and Calibration:

11.2.1 Instrument Operation

11.2.1.1 Set up the instrument with the proper operating parameters. Background correction and inter-element correction factors must be programmed into the ICP computer and applied to all analyses. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

11.2.1.2 The analyst should follow the instructions provided by the instrument manufacturer.

11.2.2 Instrument Calibration: Instrument calibration must be performed daily or each time the instrument is set up. Profile and calibrate the ICP system according to the manufacturer's instructions using the mixed calibration standard solutions and the calibration blank. Flush the system between each standard in the sequence. Use the average intensity of multiple exposures for the standardization and sample

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analysis. The calibration curve for each metal consists of a blank and a minimum of three standards.

11.3 Sample Analysis

11.3.1 Flush the system with the calibration blank solution for at least 1 minute preceding sample analysis. Analyze the calibration verification standard and the calibration blank after each 10 samples.

11.3.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established. Dilute and reanalyze samples with yttrium recoveries outside the acceptance limits of 70-130%.

11.3.3 Dilutions of High Levels of Elements of Interest: For Method 200.7, measurements for all target elements must fall within 90% of the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed 90% of the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid strength.

11.3.4 Dilutions for High Levels of Interfering Elements: Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted to a level at or below the working range.

12.0 DATA GENERATION AND CALCULATIONS

12.1 Data Generation: The data to be generated and recorded are: data analyzed, analyst, method/SOP number, standard IDs, known standard and spike concentration values, sample volume or weight, dilution factors, and the measured concentrations for samples and QC parameters.

12.2 Calibration Calculations: Calibration calculations are performed automatically by the computer workstation.

12.3 Concentration Calculations: Concentration values are calculated based on the sample volume or weight and the average of the multiple exposures for each sample. Dilution factors must be applied for samples that are diluted. All results should be reported with up to three significant figures.

12.4 Quality Control Calculations

12.4.1 Relative Percent Difference for Duplicates

$$RPD = \frac{|A - B|}{\frac{1}{2}(A + B)} \times 100\% = \frac{|A - B|}{(A + B)} \times 200\%$$

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Where: A = concentration of analyte in original sample
B = concentration of analyte in duplicate sample

12.4.2 Percent Recovery of Standards

$$\text{Percent Recovery} = \frac{\text{Analyzed Value}}{\text{Known Value}} \times 100\%$$

12.4.3 Percent Recovery of Matrix Spike

$$\text{Percent Recovery} = \frac{\text{SSC - SC}}{\text{SA}} \times 100\%$$

Where: SSC = Spiked Sample Concentration
SC = Sample Concentration (previously determined)
SA = Spike Added Concentration

12.5 Sample Calculation

12.5.1 Aqueous Sample

The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V1	=	Final volume in liters after sample preparation
V2	=	Initial volume of sample digested in liters

12.5.2 Soil Sample

The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C	=	Concentration (mg/L) from instrument readout
D	=	Instrument dilution factor
V	=	Final volume in liters after sample preparation
W	=	Weight in Kg of wet sample digested
S	=	Percent solids/100

NOTE: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the “S” factor should be omitted from the above equation.

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13.0 DATA ASSESSMENT AND CORRECTIVE ACTION

13.1 Data Assessment

13.1.1 The analyst does primary data review at the time of analysis.

13.1.2 The section supervisor, senior analyst, or designee does secondary data review

13.1.3 The section supervisor, project manager, QA manager, or laboratory director does completeness review and approval.

13.2 Corrective Action and Contingencies for Handling Out of Control Data: If any QC acceptance limit is exceeded, notify the section supervisor or QA manager, initiate a nonconformance memo (NCM) and take corrective action as advised.

14.0 DATA REDUCTION AND REPORTING

14.1 Data Reduction: The analytical value is directly transferred from the instrument into TALS. In TALS, the analytical value is converted to the reported value by the application of the appropriate calculation factors to account for the sample preparation, dilution and dry weight.

14.2 Raw Data Entry and Reporting:

14.2.1 All information pertaining to sample preparation and analysis must be entered as it is acquired in the designated bound logbook or computer generated run logs and instrument printout. This includes the test method; analyte(s); matrix; full ID for samples, standards, and QC; reagent IDs, sample volumes and weights (including units); dilution factors; analyst's initials; and date/time of preparation and analysis. All information and data pertinent to the method and analysis should be recorded to facilitate data validation. Any information which is important or may influence the test results should be noted, such as sample appearance, problems encountered, unusual conditions, reasons for repeated tests or rejected data, etc.

14.2.2 Instrument printouts must be initialed by the instrument operator.

14.2.3 Instrument printouts must be uniquely identified and maintained according to the laboratory's procedure for data storage. The computer generated spreadsheets or graphs should be organized by date and instrument and kept on file.

14.2.4 Any data that does not meet the method/project requirements or QC specifications must be documented in a NCM. Document the nonconformance, the sample(s) and parameter(s) affected, and the corrective action taken. The NCM is used to generate a case narrative to include with the final data report.

15.0 METHOD PERFORMANCE

15.1 Refer to Tables 1-9 of EPA Method 200.7 for performance data.

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15.2 Refer to Tables 4-6 of EPA Method 6010B for performance data.

16.0 POLLUTION CONTROL

16.1 It is Eurofins Xenco's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

17.0 WASTE MANAGEMENT

17.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

17.2 Waste Stream Produced by the Method: All samples and standards are placed into the laboratory acid waste stream for disposal.

18.0 REFERENCES / CROSS-REFERENCES

18.1 U.S. Environmental Protection Agency, Method 200.7, Revision 4.4.

18.2 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 6010B (Revision 2, December 1996).

18.3 CC-QAM, Eurofins Xenco Quality Assurance Manual, current revision

18.4 Associated SOPs, current revisions.

18.4.1 CC-GLO-014, Maintenance and Calibration of Balances and Class I Weights, current revision.

18.4.2 CC-GLO-015, Thermometer Calibration, current revision.

18.4.3 CC-GLO-016, Calibration and Verification of Pipettes and Dispenser, current revision.

18.4.4 CC-GLO-017, Sample Composition, Homogenization and Subsampling, current revision.

19.0 DOCUMENTATION

19.1 Analysis: The preparation of samples and standards for metals is documented in TALS.

19.2 Traceability of standards: Upon receipt or preparation, each chemical salt, solvent, acid, standard, or other reagent is entered into TALS and issued a unique ID# based upon the type and sequential order in which the item was received. Further information entered into the database includes the manufacturer, lot # (if applicable), date received or prepared,

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expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., metals ICV solution, CCV, or LCS…). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

20.0 TABLE AND ASSOCIATED DOCUMENTS

Table 1: Analytes and Reporting Limits

Table 2: LCS and MS/MSD Concentrations

Table 3: Summary of quality control requirements

Attachment 1: MSA Guidance

20.1 Associated Documents

20.1.1 Method Detection Limit Study

21.0 REVISION HISTORY

Historical File:		
	Revision 00: 10/11/02	Revision 01: 09/09/04
	Revision 02: 09/12/06	Revision 03: 08/31/09
	Revision 04: 10/08/10	Revision 05: 01/20/11
	Revision 06: 03/31/15	Revision 07: 05/29/15
	Revision 08: 06/19/17	Revision 09: 08/15/18
	Revision 10: 11/20/20	

21.1 Revision 10, dated 20 November 2020, by Tiffany Fleming

21.1.1 Biennial review
21.1.2 Updated with Eurofins branding throughout
21.1.3 Updated wording in Section 9.8 with CCV to be analyzed after 10 samples
21.1.4 Updated Section 10.4.5 with CCB analyzed with each CCV

21.2 Revision 09, dated 15 August 2018, updated by Christina Godines:

21.2.1 Updated sections throughout to merge SOP No. CC-ATM-M016, Method 200.7, into this SOP:

- Added reference to 200.7 in Sections 1.2 and 3.5;
- Added silica exception to Section 8.1;
- Added filter pore size variance in project-specific requirements to Section 8.1.3.1;
- Updated wording in Section 5.1 for Safety and added 5.1.1.3 about UV rays;

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- Added QC reference for Method 200.7 in Section 9.0;
- Added LRB, LFB, and LFM references in Sections 9.6, 9.7, and 9.8;
- Added %R and limits specific to 200.7 in Section 9.7;
- Added Sections 7.17, 7.18, and 7.19 from 200.7 SOP;
- Added dilution detail to Sections 11.3.3 and 11.3.4;
- Added 200.7 Tables 1-9 reference in Section 15.0 Method Performance;
- Updated Acceptance Criteria in Table 3;

21.2.2 Added Sections 16.0 Pollution Control and 17.0 Waste Management;

21.2.3 Updated formatting throughout sections to match current SOP format.

21.3 Reasons for changes, Revision 7:

21.3.1 Updated IDOC requirement in Section 7.1.1

21.3.2 Updated acceptance criteria in Section 7.9.8.

Table 1: Reporting Limits for Methods 6010B and 200.7

STANDARD ANALYTE LIST AND TYPICAL REPORTING LIMIT

ELEMENT	Symbol	CAS #	Reporting Limit (mg/L) Water	Reporting Limit (mg/Kg) Soil
Aluminum	Al	7429-90-5	0.100	5.0
Antimony	Sb	7440-36-0	0.005	2.0
Arsenic	As	7440-38-2	0.010	2.0
Barium	Ba	7440-39-3	0.010	1.0
Beryllium	Be	7440-41-7	0.004	0.5
Bismuth	Bi	7740-69-9	0.020	1.0

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Boron	B	7440-42-8	0.050	10
Cadmium	Cd	7440-43-9	0.005	0.5
Calcium	Ca	7440-70-2	0.200	50
Chromium	Cr	7440-47-3	0.010	1.0
Cobalt	Co	7440-48-4	0.005	1.0
Copper	Cu	7440-50-8	0.010	2.0
Iron	Fe	7439-89-6	0.200	20
Lead	Pb	7439-92-1	0.010	0.5
Lithium	Li	7439-93-2	0.020	0.5
Magnesium	Mg	7439-95-4	0.200	20
Manganese	Mn	7439-96-5	0.010	2.5
Molybdenum	Mo	7439-98-7	0.010	2.0
Nickel	Ni	7440-02-0	0.010	2.0
Phosphorus	P	7723-14-0	0.500	50
Potassium	K	7440-09-7	0.500	100
Selenium	Se	7782-49-2	0.010	1.0
Silicon	Si	7631-86-9	0.200	20
Silica	SiO2		0.429	42.9
Silver	Ag	7440-22-4	0.005	0.5
Sodium	Na	7440-23-5	1.0	100
Strontium	Sr	7440-24-6	0.005	1.0
Thallium	Tl	7440-28-0	0.005	1.0
Tin	Sn	7440-31-5	0.040	10
Titanium	Ti	7440-32-6	0.005	1.0
Vanadium	V	7440-62-2	0.005	1.0
Zinc	Zn	7440-66-6	0.020	2.5

Note: The analytes' reporting limits are evaluated yearly and may be subject to change.

Table 2: Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	4,000	4,000
Antimony	400	400
Arsenic	400	400
Barium	400	400
Beryllium	400	400
Bismuth		
Boron	800	800
Cadmium	400	400

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Calcium	40,000	40,000
Chromium	400	400
Cobalt	400	400
Copper	400	400
Iron	40,000	40,000
Lead	400	400
Lithium	400	400
Magnesium	40,000	40,000
Manganese	4,000	4,000
Molybdenum	400	400
Nickel	400	400
Phosphorous	4,000	4,000
Potassium	40,000	40,000
Selenium	400	400
Silicon	8,000	8,000
Si (as SiO_2)	17,144	17,144
Silver	400	400
Sodium	40,000	40,000
Strontium	400	400
Thallium	200	200
Tin	400	400
Titanium	400	400
Vanadium	400	400
Zinc	400	400

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Table 3: Summary of quality control requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	**6010:** 90 - 110 % recovery. **200.7:** 95 - 105 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.15	See Section 9.15
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.15
Dilution Test	One per prep batch.	For samples > 50x MDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than RL; sample results greater than 10x the blank concentration are acceptable; samples for which the contaminant is < RL may not require redigestion or reanalysis	Rerun once in a new tube. If >RL, re-digest and reanalyze samples. Note exceptions under criteria section.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action

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Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	**6010:** 80 – 120 % recovery. **200.7:** 85 – 115 % recovery. Samples for which the contaminant is < RL and the LCS results are > 120% for 6010, or > 115% for 200.7, may not require redigestion or reanalysis	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	Every 10%	75-125% recovery	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	Every 10%	75-125 % recovery; RPD ≤ 20%	See Corrective Action for Matrix Spike.

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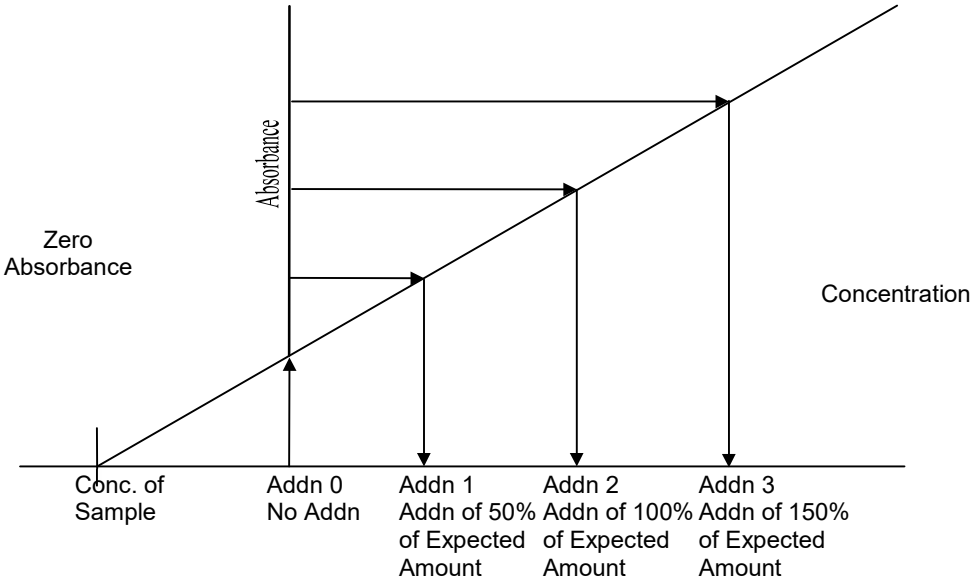
Attachment 1: MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear (r=0.995 or greater) over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

UFP-QAPP Addendum Appendix F

Field Forms

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WATER QUALITY METER INITIAL DAILY FIELD CALIBRATION FORM

YSI 3800
pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, ORP, AND DISSOVED OXYGEN

CALIBRATION
DATE:
METER ID:
SONDE ID:

pH CALIBRATION
Time: _____

pH STANDARD	PRE-CALIBRATION READING	POST-CALIBRATION READING (calibration requirement: within 0.2 pH unit of standard)
4.0		
7.0		
10.0		

CONDUCTIVITY CALIBRATION
Time: _____

CONDUCTIVITY STANDARD	PRE-CALIBRATION READING	POST-CALIBRATION READING (calibration requirement: within 10% of standard)
1.413		

TURBIDITY CALIBRATION
Time: _____

TURBIDITY STANDARD	PRE-CALIBRATION READING	POST-CALIBRATION READING (calibration requirement: within 10% of standard)
0 NTU		
126 NTU		

ORP CALIBRATION
Time: _____

ORP STANDARD	PRE-CALIBRATION READING	POST-CALIBRATION READING (calibration requirement: within 20 mV of standard)
240 millivolts		

DISSOLVED OXYGEN CALIBRATION
Time: _____

DO STANDARD	PRE-CALIBRATION READING	POST-CALIBRATION READING (calibration requirement: within <95% of standard)
100% AIR SATURATION		

COMMENTS

_____

_____

_____

SIGNATURE _____

WATER QUALITY METER DAILY CALIBRATION CHECK FIELD FORM

YSI 3800
pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, ORP, AND DISSOVED OXYGEN

CALIBRATION
DATE:
METER ID:
SONDE ID:

pH CALIBRATION CHECK
Time: _____

pH STANDARD	CALIBRATION CHECK READING (if not within 0.2 pH unit of standard, recalibrate)	RECALIBRATION REQUIRED? (Y/N)	POST-RE-CALIBRATION READING
4.0			
7.0			
10.0			

CONDUCTIVITY CALIBRATION CHECK
Time: _____

CONDUCTIVITY STANDARD	CALIBRATION CHECK READING (if not within 10% of standard, recalibrate)	RECALIBRATION REQUIRED? (Y/N)	POST-RE-CALIBRATION READING
1.413			

TURBIDITY CALIBRATION CHECK
Time: _____

TURBIDITY STANDARD	CALIBRATION CHECK READING (if not within 10% of standard, recalibrate)	RECALIBRATION REQUIRED? (Y/N)	POST-RE-CALIBRATION READING
0 NTU			
126 NTU			

ORP CALIBRATION CHECK
Time: _____

ORP STANDARD	CALIBRATION CHECK READING (if not within 20 mV of standard, recalibrate)	RECALIBRATION REQUIRED? (Y/N)	POST-RE-CALIBRATION READING
240 millivolts			

DISSOLVED OXYGEN CALIBRATION CHECK
Time: _____

DO STANDARD	CALIBRATION CHECK READING (if <95% of standard, recalibrate)	RECALIBRATION REQUIRED? (Y/N)	POST-RE-CALIBRATION READING
100% AIR SATURATION			

COMMENTS

_____

_____

SIGNATURE _____

WATER QUALITY METER END OF DAY CALIBRATION CHECK FIELD FORM

YSI 3800
pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, ORP, AND DISSOVED OXYGEN

CALIBRATION
DATE:
METER ID:
SONDE ID:

pH CALIBRATION CHECK
Time: _____

pH STANDARD	END OF DAY CALIBRATION READING	PASS/FAIL? (if not within 0.2 pH unit of standard, fail)
4.0		
7.0		
10.0		

CONDUCTIVITY CALIBRATION CHECK
Time: _____

CONDUCTIVITY STANDARD	END OF DAY CALIBRATION READING	PASS/FAIL? (if not within 10% of standard, fail)
1.413		

TURBIDITY CALIBRATION CHECK
Time: _____

STANDARD	END OF DAY CALIBRATION READING	PASS/FAIL? (if not within 10 % of standard, fail)
0 NTU		
126 NTU		

ORP CALIBRATION CHECK
Time: _____

STANDARD	END OF DAY CALIBRATION READING	PASS/FAIL? (if not within 20 mV of standard, fail)
240 millivolts		

DISSOLVED OXYGEN CALIBRATION CHECK
Time: _____

STANDARD	END OF DAY CALIBRATION READING	PASS/FAIL? (if <95% of standard, fail)
100% AIR SATURATION		

COMMENTS

_____

_____

_____

_____

_____

SIGNATURE _____

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UFP-QAPP Addendum Appendix G

Supplemental Ecological Risk Assessment Information

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Appendix G

Supplemental Ecological Risk Assessment Information

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G1

Ecological Screening Values for Surface Water and Sediment

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Surface Water Screening Values (EPA 2018)

		Freshwater Screening Values (µg/L)			Saltwater Screening Values (µg/L)		
CAS No.	Analyte	Chronic	Acute	Source	Chronic	Acute	Source
Inorganic Compounds							
Metals							
7429-90-5	Aluminum (pH 6.5 -9.0)	87	750	a	1,500		d
7440-36-0	Antimony	190	900	b	4,300		d
7440-38-2	Arsenic (filtered and unfiltered) ^	150	340	a	36	69	a
22541-54-4	Arsenic III (unfiltered) ^	148	340	b	36	69	f
7440-39-3	Barium	220	2,000	b	4	110	a
7440-41-7	Beryllium	11	93	b	0.13		d
7440-42-8	Boron	7,200	34,000	b	1,000		l
7740-43-9	Cadmium (filtered) ^ *	0.72	0.94	a	7.9	33	a
7440-70-2	Calcium	116,000		c			
16065-83-1	Chromium III (filtered) ^ *	74	323	a	103	515	s
18540-29-9	Chromium VI (filtered) ^	11	16	a	50	1,100	a
7440-48-4	Cobalt	19	120	b			
7740-50-8	Copper (filtered) ^ *	4.95	7.0	a	3.1	4.8	a
7439-89-6	Iron	1,000		a	300		d
7439-92-1	Lead (filtered) ^ *	2.5	30.1	a	8.1	210	a
7439-93-2	Lithium	440	910	b			
7439-95-4	Magnesium	82,000		c			
7439-96-5	Manganese	93	1,680	b	100		d
7439-97-6	**Mercury (filtered & unfiltered) ^ (aquatic)**	0.77	1.4	a	0.94	1.8	a
7439-97-6	**Mercury (wildlife based)**	0.0013	0.012	b, a	0.94	1.8	a
22967-92-6	**Methylmercury (aquatic life)**	0.0028	0.099	c			
7439-98-7	Molybdenum	800	7,200	b			
7440-02-0	Nickel (filtered) ^ *	52	260	a	8.2	74	a
7723-14-0	Phosphorus (elemental)	1,000		m	100		d
7440-09-7	Potassium	53,000		c			
7782-49-2	**Selenium (unfiltered) ^ (aquatic) ****	5	20	a, aa	71	290	a
7740-22-4	Silver (filtered) ^ *	1.6	0.98	b, a	0.1	1.9	e, a
7440-23-5	Sodium	680,000		c			
7440-24-6	Strontium	5,300	48,000	b			
7440-28-0	Thallium	6	54	b	6.3	710	d, g
7440-31-5	Tin	180	1,600	b			
7440-61-1	Uranium	2.6	46	c			
7440-62-2	Vanadium	27	79	b			
7740-66-6	Zinc (filtered) ^ *	120	66	a	81	90	a
7440-67-7	Zirconium	17	310	c			
Other Inorganics							
16887-00-6	Chloride	230,000	860,000	a, a			
7782-50-5	Chlorine	11	19	a, a	7.5	13	a, a
57-12-5	Cyanide (free)	5.2	22	a, a	1	1	a, a
16984-48-8	Fluoride	2,700	9,800	b, b	5,000		d
7783-06-4	Hydrogen sulfide (S_2^-, HS^-)	2	3.2	a, n	2		a
14265-45-3	Sulfite	200		b			

Notes:

From EPA. 2018. Region 4 Ecological Risk AssessmentSupplemental Guidance. March. Update.

Red font indicates a bioaccumulative chemical.

a - National Recommended Water Quality Criteria http://water.epa.gov/scitech/swguidance/standards/criteria/current/index.cfm

b - Great Lakes Initiative (GLI) Clearinghouse resources Tier II criteria revised 2013 http://www2.epa.gov/gliclearinghouse/

c - Suter, G.W. II, and Tsao, C.L. 1996. Toxicological benchmarks for screening potential contaminants of concern for effects on aquatic biota: 1996 Revision. ES/ER/TM-96/R2. https://rais.ornl.gov/documents/tm96r2.pdf

d - Florida State Criteria for Surface Water Quality Classifications http://www.dep.state.fl.us/legal/Rules/shared/62-302/302-Table.pdf

e - North Carolina Water Quality Standards https://www.epa.gov/sites/production/files/2014-12/documents/nc-classifications-wqs.pdf

f - Georgia Department of Natural Resources (GADNR) Water Use Classifications and Water Quality Standards https://epd.georgia.gov/georgia-water-qualitystandards

g - Hawaii Department of Health (HDOH) Environmental Action Levels, Chronic and Acute Surface Water (Aquatic Habitat) Standards http://ehaweb.doh.hawaii.gov/eha-cma/Leaders/HEER/environmental-hazard-evaluation-and-environmental-action-levels

l - New York Ambient Water Quality Criteria and Guidance Values: http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

m- New Jersey Department of Environmental Protection (NJDEP) Ecological Screening Criteria: http://www.nj.gov/dep/srp/guidance/ecoscreening/esc_table.pdf

^ - Screening value is for filtered (dissolved) metals. A conversion factor (CF) was used to convert the screening value for total metals in surface water to a screening value for dissolved metals in surface water. CMC (dissolved) = CMC (total) × CF. See Table 1c for screening

* - The freshwater screening value is hardness dependent. The screening value shown is for dissolved metals assuming a hardness of 50 mg/L as CaCO3. A correction for site-specific hardness was based on equations listed on the following table. If hardness data are unavailable hardness may be estimated as: H = 2.497 × Ca (mg/L) + 4.118 × Mg (mg/L).

** - Selenium concentrations in water do not reflect dietary sources to aquatic life and screening against these numbers may not be adequately protective.

EPA Region III BTAG
FRESHWATER SCREENING BENCHMARKS
7/2006

CAS#	Analyte	Screening Value (ug/l)	Ref	End Note	Bioaccumulative[q] Class of Compound	B
71-55-6	1,1,1-Trichloroethane	11	a	1	Volatile	
79-34-5	1,1,2,2-Tetrachloroethane	610	a	1	Volatile	
127-18-4	1,1,2,2-Tetrachloroethylene (PCE)	111	b	2	Volatile	
79-00-5	1,1,2-Trichloroethane	1200	a	1	Volatile	
79-01-6	1,1,2-Trichloroethene (TCE)	21	b	2		
92-52-4	1,1-Biphenyl	14	c	1	PAH	
75-34-3	1,1-Dichloroethane	47	c	1	Volatile	
75-35-4	1,1-Dichloroethene (1,1-Dichloroethylene)	25	a	1	Volatile	
75-35-4	1,1-Dichloroethylene	25	a	1	Volatile	
634-66-2	1,2,3,4-Tetrachlorobenzene	1.8	b	2	Other Semi-Volatile	B
87-61-6	1,2,3-Trichlorobenzene	8	b	2	Other Semi-Volatile	
95-94-3	1,2,4,5-Tetrachlorobenzene	3	d	3	Other Semi-Volatile	B
120-82-1	1,2,4-Trichlorobenzene	24	b	2	Volatile	B
95-63-6	1,2,4-Trimethylbenzene	33	e,f	4	Volatile	B
95-50-1	1,2-Dichlorobenzene	0.7	b	2	Volatile	B
107-06-2	1,2-Dichloroethane	100	b	2	Volatile	
540-59-0	1,2-Dichloroethene (1,2-Dichloroethylene)	590	a	1	Volatile	
540-59-0	1,2-Dichloroethylene	590	a	1	Volatile	
156-60-5	1,2-Trans-Dichloroethylene	970	g	5	Volatile	
108-67-8	1,3,5-Trimethylbenzene	71	h,i	6	Volatile	
541-73-1	1,3-Dichlorobenzene	150	b	2	Volatile	B
542-75-6	1,3-Dichloropropene (1,3-Dichloropropylene)	0.055	a	1	Volatile	
542-75-6	1,3-Dichloropropylene	0.055	a	1	Volatile	
106-46-7	1,4-Dichlorobenzene	26	b	2	Volatile	B
99-08-1	1-Methyl-3-nitrobenzene	750	h,i	6	Other Semi-Volatile	
99-99-0	1-Methyl-4-nitrobenzene (4-Nitrotoluene)	1900	h,i	6	Other Semi-Volatile	
90-12-0	1-Methylnaphthalene	2.1	a	1	PAH	
71-41-0	1-Pentanol	110	a	1	Volatile	
10222-01-2	2,2-Dibromo-3-nitrilopropionamide	20	e,f	4,7	Other Semi-Volatile	
58-90-2	2,3,4,6-Tetrachlorophenol	1.2	d	3	Other Semi-Volatile	
1746-01-6	2,3,7,8-TCDD-Dioxin	3.1E-09	j	8	Dioxin/Furans	B
51207-31-9	2,3,7,8-TCDF		k	9	Dioxin/Furans	B
93-72-1	2,4,5-TP (Silvex)	30	d	3	Volatile	
93-76-5	2,4,5-Trichlorphenoxyacetic acid	686	l,m	10	Phenoxycaceticacid Herbicide	
88-06-2	2,4,6-Trichlorophenol	4.9	g	5	Other Semi-Volatile	
118-96-7	2,4,6-Trinitrotoluene (TNT)	100	h,i	6	Other Semi-Volatile	
120-83-2	2,4-Dichlorophenol	11	g	5	Other Semi-Volatile	
121-14-2	2,4-Dinitrotoluene	44	g	5	Other Semi-Volatile	
606-20-2	2,6-Dinitrotoluene	81	g	5	Other Semi-Volatile	
35572-78-2	2-Amino-4,6-dinitrotoluene	1480	h,i	6	Other Semi-Volatile	
78-93-3	2-Butanone	14000	a	1	Volatile	
95-57-8	2-Chlorophenol	24	d	3	Other Semi-Volatile	
591-78-6	2-Hexanone	99	a	1	Volatile	
91-57-6	2-Methylnaphthalene	4.7	e,f	4	PAH	
95-48-7	2-Methylphenol	13	a	1	Other Semi-Volatile	
88-75-5	2-Nitrophenol	1920	h,i	6	Other Semi-Volatile	
111-13-7	2-Octanone	8.3	a	1	Other Semi-Volatile	

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67-63-0	2-Propanol	7.5	a	1	Volatile	
91-94-1	3,3'- Dichlorobenzidine	4.5	d	3	Other Semi-Volatile	
99-08-1	3-Nitrotoluene (1-Methyl-3-nitrobenzene)	750	h,i	6	Other Semi-Volatile	
106-68-3	3-Octanone	3571	a	11	Volatile	
101-55-3	4-Bromophenyl phenyl ether	1.5	c	1	Other Semi-Volatile	B
106-47-8	4-Chloroaniline	232	l,m	10	Other Semi-Volatile	
7005-72-3	4-Chlorophenyl- phenyl ether				Other Semi-Volatile	B
99-87-6	4-Isopropyltoluene (Cymene)	85	h,i	6	Other Semi-Volatile	
108-10-1	4-Methyl-2-pentanone	170	a	1	Volatile	
106-44-5	4-Methylphenol	543	h,i	6	Other Semi-Volatile	
100-02-7	4-Nitrophenol	60	a	12	Other Semi-Volatile	
99-99-0	4-Nitrotoluene	1900	h,i	6	Other Semi-Volatile	
83-32-9	Acenaphthene	5.8	b	2	PAH	B
208-96-8	Acenapthylene				PAH	B
67-64-1	Acetone	1500	a	1	Volatile	
75-05-8	Acetonitrile	12000	g	5	Volatile	
309-00-2	Aldrin	3	n	13	Organochlorine Pesticide	B
7429-90-5	Aluminum	87	n	13	Inorganic/Metal	
7664-41-7	Ammonia (un-ionized)	19	b	2	Inorganic	
62-53-3	Aniline	2.2	b	2	Other Semi-Volatile	
120-12-7	Anthracene	0.012	b	2	PAH	B
7440-36-0	Antimony	30	a	1	Inorganic/Metal	
140-57-8	Aramite	3.09	l,m	10	Other Semi-Volatile	
12674-11-2	Aroclor 1016 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
11104-28-2	Aroclor 1221 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
11141-16-5	Aroclor 1232 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
53469-21-9	Aroclor 1242 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
12672-29-6	Aroclor 1248 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
11097-69-1	Aroclor 1254 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
11096-82-5	Aroclor 1260 (total PCBs)	0.000074	j	8	Other Pesticide/PCB	B
7440-38-2	Arsenic	5	b	2	Inorganic/Metal	B
22569-72-8	Arsenic III	55	a	14	Inorganic/Metal	
17428-41-0	Arsenic V	3.1	a	1	Inorganic/Metal	
1912-24-9	Atrazine	1.8	b	2	Triazine Herbicide	
86-50-0	Azinophosmethyl (Guthion)	0.01	n	13	Organophosphorus Pesticide	
7440-39-3	Barium	4	a	1	Inorganic/Metal	
71-43-2	Benzene	370	b	2	Volatile	
92-87-5	Benzidine	3.9	a	1	Other Semi-Volatile	
56-55-3	Benzo(a)anthracene	0.018	b	2	PAH	B
50-32-8	Benzo(a)pyrene	0.015	b	2	PAH	B
205-99-2	Benzo(b)fluoranthene				PAH	B
191-24-2	Benzo(g,h,i)perylene				PAH	B
207-08-9	Benzo(k)fluoranthene				PAH	B
65-85-0	Benzoic Acid	42	a	1	Other Semi-Volatile	
100-51-6	Benzyl alcohol	8.6	a	1	Other Semi-Volatile	
7440-41-7	Beryllium	0.66	a	1	Inorganic/Metal	
	BHC (non Lindane)	2.2	a	1	Organochlorine Pesticide	

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319-84-6	BHC, alpha				Organochlorine Pesticide	B
319-85-7	BHC, beta				Organochlorine Pesticide	B
319-86-8	BHC, delta	141	h,i	6	Organochlorine Pesticide	B
58-89-9	BHC, gamma (Lindane)	0.01	b	2	Organochlorine Pesticide	B
92-52-4	Biphenyl (1,1-Biphenyl)	14	c	1	Other Semi-Volatile	
117-81-7	bis (2-ethylhexyl) phthalate	16	b	2	Other Semi-Volatile	
7440-42-8	Boron	1.6	a	1	Inorganic/Metal	
75-25-2	Bromoform	320	c	1	Volatile	
74-83-9	Bromomethane				Volatile	
85-68-7	Butyl benzyl phthalate	19	c	1	Other Semi-Volatile	
7440-43-9	Cadmium (hardness=100)	0.25	n	15	Inorganic/Metal	B
7440-70-2	Calcium	116000	a	16	Inorganic/Metal	
63-25-2	Carbaryl (Sevin)	0.2	b	2	Other Pesticide/PCB	
1563-66-2	Carbofuran	1.8	b	2	N-Methylcarbamate herbicide	
75-15-0	Carbon disulfide	0.92	a	1	Volatile	
56-23-5	Carbon tetrachloride	13.3	b	2	Volatile	
57-74-9	Chlordane	0.0022	n	13	Organochlorine Pesticide	B
16887-00-6	Chloride	230000	n	13	Inorganic/Metal	
7782-50-5	Chlorine (TRC)	11	n	13	Inorganic/Metal	
108-90-7	Chlorobenzene	1.3	b	2	Volatile	
510-15-6	Chlorobenzilate	7.16	l,m	10	Other Pesticide/PCB	
67-66-3	Chloroform	1.8	b	2	Volatile	
2921-88-2	Chloropyrifos	0.0035	b	2	Organophosphorus Pesticide	B
16065-83-1	Chromium III (hardness=100)	74	n	15	Inorganic/Metal	
7440-47-3	Chromium Total (hardness=100)	85	n		Inorganic/Metal	
18540-29-9	Chromium VI (hardness=100)	11	n		Inorganic/Metal	B
218-01-9	Chrysene				PAH	B
7440-48-4	Cobalt	23	a	1	Inorganic/Metal	
7440-50-8	Copper (hardness=100)	9	n	13,15	Inorganic/Metal	B
98-82-8	Cumene	2.6	e,f	4	Volatile	
57-12-5	Cyanide, free	5	b	2	Inorganic/Metal	
99-87-6	Cymene	85	h,i	6	Volatile	
72-54-8	DDD (p,p')	0.011	a	1	Organochlorine Pesticide	B
72-55-9	DDE				Organochlorine Pesticide	B
50-29-3	DDT (4,4' DDT)	0.0005	n	13	Organochlorine Pesticide	B
	DDT (op + pp)				Organochlorine Pesticide	B
	DDT, Total				Organochlorine Pesticide	B
	DDT/DDE/DDD (total)	0.000011	j	8	Organochlorine Pesticide	B
124-18-5	Decane	49	a	1	Other Semi-Volatile	
8065-48-3	Demeton	0.1	n	13	Other Semi-Volatile	
117-81-7	Di(2-ethylhexyl)phthalate (Bis(2-ethylhexyl) phthalate)	16	b	2	Other Semi-Volatile	
333-41-5	Diazinon	0.043	a	1	Other Pesticide/PCB	B
53-70-3	Dibenzo(a,h)anthracene				PAH	B
132-64-9	Dibenzofuran	3.7	a	1	Other Semi-Volatile	
3252-43-5	Dibromoacetonitrile	20	e,f	4,7	Other Semi-Volatile	
25321-22-6	Dichlorobenzene (mixed isomers)	5	e,f	4,17	Volatile	
75-09-2	Dichloromethane	98.1	b	2	Volatile	

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SEQ NO-35-8	Dichlorophenols, total	0.2	b	2	Other Semi-Volatile	
542-75-6	Dichloropropene (1,3-Dichloropropylene)	0.055	a	1	Volatile	
115-32-2	Dicofol (Kelthane)				Other Pesticide/PCB	B
60-57-1	Dieldrin	0.056	n	13	Organochlorine Pesticide	B
84-66-2	Diethylphthalate	210	a	1	Other Semi-Volatile	
60-51-5	Dimethoate	6.2	b	2	Other Pesticide/PCB	
84-74-2	Di-n-butyl phthalate	19	b	2	Other Semi-Volatile	
117-84-0	Di-n-octyl phthalate	22	h,i	6	Other Semi-Volatile	
88-85-7	Dinoseb	0.05	b	2	Other Pesticide/PCB	
117-84-0	Dioctylphthalate (Di-n-octyl phthalate)	22	h,i	6	Other Semi-Volatile	
298-04-4	Disulfoton				Pesticide	B
115-29-7	Endosulfan (alpha and beta)	0.02	b	2	Organochlorine Pesticide	B
959-98-8	Endosulfan I (a-endosulfan)	0.051	c	1	Organochlorine Pesticide	B
33213-65-9	Endosulfan II (b-endosulfan)	0.051	c	1	Organochlorine Pesticide	B
72-20-8	Endrin	0.036	n	13	Organochlorine Pesticide	B
100-41-4	Ethylbenzene	90	b	2	Volatile	
107-06-2	Ethylene dichloride (1,2-Dichloroethane)	100	b	2	Volatile	
107-21-1	Ethylene glycol	192000	b	2	Other Semi-Volatile	
206-44-0	Fluoranthene	0.04	b	2	PAH	B
86-73-7	Fluorene	3	b	2	PAH	B
16984-48-8	Fluoride (hardness = 100)	2119.4	e,f	4,15	Inorganic	
7782-41-4	Fluorine	1080	a	12	Inorganic	
86-50-0	Guthion	0.01	n	13	Other Pesticide/PCB	
319-86-8	HCH, d- (BHC, delta)	141	h,i	6	Organochlorine Pesticide	
58-89-9	HCH, gamma (Lindane) (BHC, gamma)	0.01	b	2	Organochlorine Pesticide	
76-44-8	Heptachlor	0.0019	n	13	Organochlorine Pesticide	B
	Heptachlor & Heptachlor epoxide				Organochlorine Pesticide	B
1024-57-3	Heptachlor epoxide	0.0019	n	13	Organochlorine Pesticide	B
118-74-1	Hexachlorobenzene	0.0003	d	18	Other Semi-Volatile	B
87-68-3	Hexachlorobutadiene	1.3	b	2	Volatile	B
608-73-1	Hexachlorocyclohexanes (HCH, BHC)	0.01	b	2	Organochlorine Pesticide	B
77-47-4	Hexachlorocyclopentadiene				Organochlorine Pesticide	B
67-72-1	Hexachloroethane	12	c	1	Volatile	B
110-54-3	Hexane	0.58	a	1	Volatile	
2691-41-0	HMX (Octogen)	150	h,i	6	Other Pesticide/PCB	
302-01-2	Hydrazine (hardness <50)	5	e,f	4	Volatile	
302-01-2	Hydrazine (hardness =>50)	10	e,f	4	Volatile	
123-31-9	Hydroquinone	2.2	e,f	4	Other Semi-Volatile	
193-39-5	Indeno(1,2,3-c,d)pyrene				PAH	B
7439-89-6	Iron	300	b	2	Inorganic/Metal	
29761-21-5	Isodecyl diphenyl phosphate	1.7	e,f	4	Other Semi-Volatile	
98-82-8	Isopropylbenzene (Cumene)	2.6	e,f	4		
	Isothiazolones, total	1	e,f	4	Other Semi-Volatile	
7439-92-1	Lead (hardness=100)	2.5	n	13,15	Inorganic/Metal	B
58-89-9	Lindane (BHC,gamma)	0.01	b	2	Organochlorine Pesticide	
	Linear alkyl benzene sulfates (LAS)	40	e,f	4,19	Other Semi-Volatile	
7439-93-2	Lithium	14	a	1	Inorganic/Metal	

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7439-95-4	Magnesium	82000	a	16	Inorganic/Metal	
121-75-5	Malathion	0.097	c	1	Other Pesticide/PCB	
7439-96-5	Manganese	120	a	1	Inorganic/Metal	
7439-97-6	Mercury	0.026	b	2	Inorganic/Metal	
72-43-5	Methoxychlor	0.019	c	1	Organochlorine Pesticide	B
78-93-3	Methyl ethyl ketone (2-Butanone)	14000	a	1	Volatile	
111-13-7	Methyl hexyl ketone (2-Octanone)	8.3	a	1	Volatile	
108-10-1	Methyl isobutyl ketone (4-Methyl-2-pentanone)	170	a	1	Volatile	
80-62-6	Methyl methacrylate	2800	l,m	10	Volatile	
1634-04-4	Methyl tert-butyl ether (MTBE)	11070	h,i	6	Other Semi-Volatile	
6317-18-6	Methylene bisthiocyanate	1	e,f	4	Other Semi-Volatile	
75-09-2	Methylene chloride (Dichloromethane)	98.1	b	2	Volatile	
22967-92-6	Methylmercury	0.004	b	2	Volatile	B
2385-85-5	Mirex	0.001	n	13	Chlorinated Pesticides	B
7439-98-7	Molybdenum	73	b	2	Inorganic/Metal	
108-90-7	Monochlorobenzene (Chlorobenzene)	1.3	b	2		
	Monochlorophenols, total	7	b	2	Other Semi-Volatile	
91-20-3	Naphthalene	1.1	b	2	PAH	
84-74-2	n-Butylphthalate (Di-n-butyl phthalate)	19	b	2	Other Semi-Volatile	
7440-02-0	Nickel (hardness=100)	52	n	13,15	Inorganic/Metal	B
139-13-9	Nitrilotriacetic acid	5000	e,f	4,20		
	Nitrite (cold water)	20	e,f	4	Anion	
	Nitrite (warm water)	100	e,f	4	Anion	
55-63-0	Nitroglycerine	138	h,i	6	Other Semi-Volatile	
55-18-5	N-Nitrosodiethylamine	768	l,m	10	Other Semi-Volatile	
62-75-9	N-Nitrosodimethylamine	117	o	21	Other Semi-Volatile	
86-30-6	N-Nitrosodiphenylamine	210	a	1	Other Semi-Volatile	
95-48-7	o-Cresol (2-Methylphenol)	13	a	1	Other Semi-Volatile	
56-38-2	Parathion	0.013	n	13	PAH	
	Parathion and methyl parathion	0.008	e,f	4	Organophosphorus Pesticide	
1336-36-3	PCBs, total	0.000074	j	8	Other Pesticide/PCB	
106-44-5	p-Cresol (4-Methylphenol)	543	h,i	6	Other Semi-Volatile	
608-93-5	Pentachlorobenzene	6	b	2	Other Semi-Volatile	B
76-01-7	Pentachloroethane	56.4	l,m	10	Other Semi-Volatile	
87-86-5	Pentachlorophenol (pH = 7.8)	0.5	b	2	Other Semi-Volatile	B
78-11-5	PETN (Pentaerythrite-tetranitrate)	85000	h,i	6	Other Semi-Volatile	
	pH	6.5-9	b	2	Inorganic	
85-01-8	Phenanthrene	0.4	b	2	PAH	B
108-95-2	Phenol	4	b	2	Other Semi-Volatile	B
100-42-5	Phenylethylene	72	b	2	Other Semi-Volatile	
298-02-2	Phorate	3.62	l,m	10		
7723-14-0	Phosphorus		p	22	Inorganic/Metal	
51207-31-9	Polychlorinated dibenzofurans		k	9	Dioxins/Furans	
	Polychlorinated dibenzo-p-dioxins (PCDD-S)		k	9	Dioxins/Furans	
7440-09-7	Potassium	53000	a	16	Inorganic/Metal	
103-65-1	Propyl benzene	128	h,i	6	Volatile	
129-00-0	Pyrene	0.025	b	2	PAH	B

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110-86-1	Pyridine	2380	l,m	10	Volatile	
	Quaternary ammonium compounds, total	10	e,f	4	Inorganic	
121-82-4	RDX (Cyclonite)	360	h,i	6	Explosive	
7782-49-2	Selenium	1	b	2	Inorganic/Metal	B
7440-22-4	Silver (hardness = 100)	3.2	n	13,15	Inorganic/Metal	B
7440-23-5	Sodium	680000	a	16	Inorganic/Metal	
7440-24-6	Strontium	1500	a	1	Inorganic/Metal	
100-42-5	Styrene (Phenylethylene)	72	b	2	Volatile	
7783-06-4	Sulfide (Hydrogen Sulfide)	2	n	13	Anion	
	Sulfite	200	e,f	4	Anion	
95-94-3	Tetrachlorobenzene (1,2,4,5-Tetrachlorobenzene)	3	d	3	Other Semi-Volatile	
79-34-5	Tetrachloroethane (1,1,2,2-Tetrachloroethane)	610	a	1	Volatile	
127-18-4	Tetrachloroethene (1,1,2,2-Tetrachloroethylene)	111	b	2	Volatile	
127-18-4	Tetrachloroethylene (1,1,2,2-Tetrachloroethylene)	111	b	2	Volatile	
56-23-5	Tetrachloromethane (Carbon tetrachloride)	13.3	b	2	Volatile	
	Tetrachlorophenols, total	1	b	2	Other Semi-Volatile	
7440-28-0	Thallium	0.8	b	2	Inorganic/Metal	
7440-31-5	Tin	73	a	1	Inorganic/Metal	
108-88-3	Toluene	2	b	2	Volatile	
8001-35-2	Toxaphene	0.0002	n	13	Organochlorine Pesticide	B
156-60-5	trans-1,2-Dichloroethylene (1,2-trans-Dichloroethylene)	970	g	5	Volatile	
75-25-2	Tribromomethane (Bromoform)	320	c	1	Volatile	
688-73-3	Tributyltin	0.008	b	2	Inorganic/Metal	B
12002-48-1	Trichlorobenzene (mixed isomers)	5	e,f	4,23	Other Semi-Volatile	
79-00-5	Trichloroethane (1,1,2-Trichloroethane)	1200	a	1	Volatile	
79-01-6	Trichloroethene (Trichloroethylene)	21	b	2	Volatile	
79-01-6	Trichloroethylene	21	b	2	Volatile	
67-66-3	Trichloromethane (Chloroform)	1.8	b	2	Volatile	
25167-82-2	Trichlorophenols, total	18	b	2	Other Semi-Volatile	
1582-09-8	Trifluralin	0.2	b	2		
115-86-6	Triphenyl phosphate	4	e,f	4	Other Semi-Volatile	
7440-61-1	Uranium (hardness = 100)	2.6	a	1	Inorganic/Metal	
7440-62-2	Vanadium	20	a	1	Inorganic/Metal	
1314-62-1	Vanadium pentoxide	15	h,i	6	Inorganic/Metal	
108-05-4	Vinyl acetate	16	a	1	Volatile	
100-42-5	Vinyl benzene (Pheylethylene)	72	b	2	Volatile	
75-01-4	Vinyl chloride	930	d	3	Volatile	
75-35-4	Vinylidene chloride (1,1-Dichloroethylene)	25	a	1	Volatile	
108-38-3	Xylene, m-	1.8	c	1	Volatile	
1330-20-7	Xylenes (total)	13	a	1	Volatile	
7440-66-6	Zinc (hardness = 100)	120	n	15	Inorganic/Metal	B
7440-67-7	Zirconium	17	a	1	Inorganic/Metal	

Note: Values are expressed in terms of dissolved analyte in the water column except for those indicated with endnote 2 which are expressed in terms of total concentration.

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[o]Oregon DEQ (Department of Environmental Quality). 1998. Guidance for Ecological Risk Assessment: Level II Screening Benchmark Values. Updated December 2001. Oregon Dept. Env. Qual., Portland. SLV-2. Available at: http://www.deq.state.or.us/wmc/documents/eco-2slv.pdf

[p]U.S. EPA. Ecoregional Nutrient Criteria. Available at: http:/www.epa.gov/waterscience/criteria/nutrient/ecoregions.

[q]U.S. EPA. 2000. Bioaccumulation Testing and Interpretation for the Purpose of Sediment Quality Assessment. Available at: http://www.epa.gov/waterscience/cs/biotesting/bioaccum.pdf

ENDNOTES

[1]GLWQI Tier II values (U.S. EPA OSWER 1996[c], Suter and Tsao 1996[a]).
[2]The Canadian Water Quality Guidelines values refer to the total concentration in an unfiltered sample.
[3] Final Chronic Value.
[4]Value calculated using Tier I or Tier II methodology as described in NYCRR (1999[f]).
[5]Data are Ohio River Basin aquatic life Tier II values from the OMZA.
[6]Values derived using LC_{50} approach in accordance with methodology defined in TNRCC (2000[i], 2001[h]). Concentrations of non-persistent toxic materials shall not exceed concentrations which are chronically toxic (0.1 of acute LC_{50} values) to the most sensitive aquatic species. Concentrations of persistent toxic materials that do not bioaccumulate shall not exceed concentrations which are chronically toxic (0.05 of LC_{50} values) to the most sensitive aquatic species. Concentrations of toxic materials that bioaccumulate shall not exceed concentrations that are chronically toxic (0.01 of LC_{50}) to the most sensitive aquatic species.
[7]The screening value of 20 ug/L is for 2,2-Dibromo-3-nitrilopropionamide and dibromoacetonitrile combined.
[8]This value is based on the food chain and not direct toxicity.
[9]Congener- and receptor-specific dioxin equivalency.
[10]Value is based on an interim criterion developed according to the procedures described in U.S. EPA Region 5 (1999[m]). Source data used in developing interim criteria were obtained through the Aquatic Toxicity Information Retrieval (ACQUIRE) database.
[11]Value is the lowest test EC_{20} for fish (Suter and Tsao 1996[a]).
[12]Value is the lowest population EC_{20} (Suter and Tsao 1996[a]).
[13]Criterion Continuous Concentration (CCC) values are used rather than Criteria Maximum Concentration (CMC) values. The CMC is an estimate of the highest concentration of a material in surface water to which an aquatic community can be exposed briefly without resulting in an unacceptable effect. The CCC is an estimate of the highest concentration of a material in surface water to which an aquatic community can be exposed indefinitely without resulting in an unacceptable effect. No CCC values are available for aldrin and silver; therefore the CMC values are used. The Criteria for chlordane, 4,4' DDT, heptachlor and heptachlor epoxide are based on 304(a) aquatic life criterion issued in 1980. The Minimum Data Requirements and derivation procedures were different in the 1980 and 1985 Guidelines. The AWQC values are divided by 2 to obtain a value that is more comparable to a CMC derived using the 1985 Guidelines.
[14]Value is the lowest sensitive species test EC_{20} for (Suter and Tsao 1996[a]).
[15] See Freshwater Values for Hardness-Dependent Contaminants table if hardness does not equal 100. AWQC (2002[n]) and Suter and Tsao (1996[a]) values were used instead of CCME (2002[b]) values if equations for variable hardness were not provided in the CCME document. The CCME values are: 0.017 ug/l cadmium, 8.9 ug/l chromium III, 1 ug/l chromium VI, 0.1 ug/l silver, and 30 ug/l zinc.
[16] Lowest Chronic Value for Daphnids (Suter and Tsao 1996[a]).
[17] Applies to the sum of 1,2-, 1,3- and 1,4-dichlorobenzene.
[18]Wildlife value.
[19]LAS with side chains greater than 13 carbons only; applies to the sum of these substances. 1996[a]
[20] Applies to nitrilotriacetate (NY DEC 1993[e]).
[21] Oregon Water Quality Criteria Freshwater acute criteria divided by 50, for acute to chronic conversion (Oregon DEQ 1998[o]).
[22] Select a value from the criteria documents (Lakes and Reservoirs, Rivers and Streams) for the ecoregion, which corresponds to the site location.
[23] Applies to the sum of 1,2,3-, 1,2,4- and 1,3,5-trichlorobenzene.

Sediment Screening Values (EPA 2018)

CAS No.	Analyte	*Freshwater Sediment Screening Value*		Source	*Marine/Estuarine Sediment Screening Value*		Source
		ESV	RSV		ESV	RSV	
Inorganic Compounds - mg/kg dry weight							
Metals - mg/kg dw							
7429-90-5	Aluminum	25,000	58,000	i			
7440-36-0	Antimony	2	25	a	2	25	a
7440-38-2	Arsenic	9.8	33	b	7.24	41.6	c
7440-39-3	Barium	20	60	b			
7440-43-9	Cadmium	1	5	b	0.68	4.21	c
7440-47-3	Chromium (Total)	43.4	111	b	52.3	160	c
7440-48-4	Cobalt	50		b			
7440-50-8	Copper	31.6	149	b	18.7	108	c
7439-89-6	Iron	20,000	40,000	g			
7439-92-1	Lead	35.8	128	b	30.2	112	c
7439-96-5	Manganese	460	1,100	g			
7439-97-6	**Mercury (inorganic - aquatic life)**	0.18	1.1	b	0.13	0.7	c
7439-97-6	**Mercury (inorganic - wildlife based)**	0.17	0.17	i			
22967-92-6	**Methylmercury (wildlife based)**	0.00045	0.0045	i			
7440-02-0	Nickel	22.7	48.6	b	15.9	42.8	c
7782-49-2	**Selenium**	0.72	2.9	i			
7782-49-2	**Selenium (wildlife)**	0.8	1.2	i			
7440-22-4	Silver	1	2.2	b	0.73	1.77	c
7440-61-1	Uranium	100	1,000	i			
7440-66-6	Zinc	121	459	b	124	271	c
Other Inorganics - mg/kg dw							
7664-41-7	Ammonia	230	300	h			
18946-25-8	Sulfides (Total)	39	61	h			

Notes:

From EPA. 2018. Region 4 Ecological Risk AssessmentSupplemental Guidance. March. Update.

ESV = Ecological Screening Value

RSV = Refinement Screening Value

Red font indicates a bioaccumulative chemical.

a - Long, Edward R., and Lee G. Morgan. 1991. The Potential for Biological Effects of Sediment-Sorbed Contaminants Tested in the National Status and Trends Program.

b - MacDonald, et al. 2003. Development and Evaluation of Numerical Sediment Quality Assessment Guidelines for Florida Inland Waters. Florida Department of Environmental Protection, Tallahassee, FL. Development and Evaluation of Numerical Sediment
Quality Assessment Guidelines for Florida Inland Waters. Used threshold effect concentration (TEC) for the ESV and probable effect concentration (PEC) for the RSV.

c - MacDonald, D.D. 1994. Approach to the Assessment of Sediment Quality in Florida Coastal Waters. Florida Department of Environmental Protection. 1994 Florida Sediment Quality Assessment Guidelines for Florida Coastal Waters.

g - Persaud, D., R. Jaagumagi and A. Hayton. 1993. Guidelines for the protection and management of aquatic sediment quality in Ontario. Ontario Ministry of the Environment. Queen's Printer of Ontario.

h - Washington State Sediment Management Standards, Cleanup Objectives.
http://www.ecy.wa.gov/programs/tcp/smu/sed_standards.htm

i - Los Alamos National Laboratory ECORISK Database. September 2017.
http://www.lanl.gov/environment/protection/eco-risk-assessment.php

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Ecological Exposure Parameters

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Exposure Parameters for Aquatic Wildlife

RIVER OTTER (PISCIVOROUS MAMMAL	Value	Unit	Notes
Body Weight	7.43	kg	EPA, 1993 average of adult male and female body weights for eastern US.
Food Ingestion Rate	0.0481	kg dry wt/kg-day	EPA, 1993 allometric equation for all mammals [Equation 3-7]
Incidental Sediment Ingestion Rate	2.00%	% of total mass of diet	Assumed, conservative estimate
Habitat Range	859	acres	EPA, 1993 average of adult male and female ranges for Texas, converted to acres
Water Ingestion Rate	0.081	L/kg-day	EPA 1993, average of male and female rates
GREAT BLUE HERON (PISCIVOROUS BIRD)			
Body Weight	2.229	kg	EPA, 1993, value for eastern North America
Food Ingestion Rate	0.0450	kg dry wt./kg-day	Converted assuming 75% prey moisture
Food Ingestion Rate	0.18	kg wet wt./kg-day	EPA, 1993
Incidental Sediment Ingestion Rate	2.00%	% of total mass of diet	Assumed, conservative estimate
Habitat Range	11	acres	EPA, 1993, average of values provided
Water Ingestion Rate	0.045	L/kg-day	EPA, 1993
MUSKRAT (HERBIVOROUS MAMMAL)			
Body Weight	1.17	kg	EPA 1993, average of adult male and female weights given throughout year
Food Ingestion Rate	0.045	kg dry wt./kg-day	EPA 1993, converted assuming 85% food moisture
Food Ingestion Rate	0.3	kg wet wt./kg-day	EPA 1993, average of greens and green and corn ingestion rates
Incidental Soil Ingestion Rate	2.00%	% of total mass of diet	Beyer et al. 1994, value for woodchuck
Habitat Range	0.33	acres	EPA, 1993, average of values provided
Water Ingestion Rate	0.975	L/kg-day	EPA 1993, average of male and female rates
MALLARD DUCK (HERBIVOROUS BIRD)			
Body Weight	1.134	kg	EPA 1993, average of adult male and female weights given throughout year
Food Ingestion Rate	0.049	kg dry wt./kg-day	Calculated using allometric equation for all birds from Nagy 2001
Incidental Soil Ingestion Rate	3.30%	% of total mass of diet	Beyer et al. 1994
Habitat Range	1075	acres	EPA 1993, average of values provided
Water Ingestion Rate	0.0565	L/kg-day	EPA 1993, average of adult male and female rates
LESSER SCAUP (INSECTIVOROUS BIRD)			
Body Weight	0.815	kg	EPA, 1993 average of male and female body weights in US
Food Ingestion Rate	0.0625	kg dry wt/kg-day	EPA, 1993 allometric equation for all birds [Equation 3-3]
Incidental Sediment Ingestion Rate	2.00%	% of total mass of diet	Assumed, conservative estimate
Habitat Range	219	acres	EPA, 1993 converted to acres
Water Ingestion Rate	0.0630	L/kg-day	EPA, 1993 average of adult male and female rates

Notes:

Food ingestion rates presented in wet weight were converted to dry weight assuming the following water content:

Plant foliage: 85% (EPA 2007)

Fish/benthic invertebrates: 75% (USACHPPM 2004)

References:

Beyer, W.N., E. Conner, and S. Gerould. 1994. Estimates of soil ingestion by wildlife. J. Wildl. Manage. 58:75−382.

U.S. Environmental Protection Agency (EPA). 1993. Wildlife Exposure Factors Handbook. 2 Volumes. EPA/630/R-93/187a. EPA Office of Research and Development, Washington, DC.

Nagy, KA. 2001. Food requirements of wild animals: predictive equations for free-living mammals, reptiles, and birds. Nutrition Abstracts and Reviews Series B 71, 21R-31R.

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Ecological Uptake Factors

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Uptake Factors for Plants, Benthos, and Fish

Analyte	Plant BAFs	Note	Benthic Invertebrate BSAFs	Note	Fish BCFs (L/Kg)[i]	Fish Uptake (mg/kg dr wt)[j]
Antimony	ln(Cp) = 0.938 * ln(Cs) - 3.233	a	Log(Cb)= 1.089+0.278[Log(Cs)]	c	100	400
Arsenic	Cp = 0.03752 * Cs	b	0.127	d	300	1200
Barium	Cp = 0.156 * Cs	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	4	16
Beryllium	ln(Cp) = 0.7345 * ln(Cs) - 0.5361	a	Log(Cb)= 1.089+0.278[Log(Cs)]	c	100	400
Cadmium	ln(Cp) = 0.546 * ln(Cs) - 0.475	b	3.073	e	200	800
Chromium	Cp = 0.041 * Cs	b	0.468	e	200	800
Cobalt	Cp = 0.0075 * Cs	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	300	1200
Copper	ln(Cp) = 0.394 * ln(Cs) + 0.668	b	Log(Cb)= 1.089+0.278[Log(Cs)]	f	200	800
Lead	ln(Cp) = 0.561 * ln(Cs) - 1.328	b	0.066	d	300	1200
Manganese	Cp = 0.079 * Cs	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	400	1600
Mercury	NA		1.136	h	1000	4000
Nickel	ln(Cp) = 0.748 * ln(Cs) - 2.223	b	Log(Cb)= -0.440+0.695[Log(Cs)]	g	100	400
Selenium	ln(Cp) = 1.104 * ln(Cs) - 0.677	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	200	800
Silver	Cp = 0.014 * Cs	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	5	20
Vanadium	Cp = 0.00485 * Cs	b	Log(Cb)= 1.089+0.278[Log(Cs)]	c	NA	NA
Zinc	ln(Cp) = 0.554 * ln(Cs) + 1.575	b	1.936	h	1000	4000

Notes:
a - Regression derived from measured data by EPA (2007)
b - Bechtel-Jacobs 1998a
c - Bechtel-Jacobs 1998b, based on regression model for copper
d - Bechtel-Jacobs 1998b, median BSAF for non-depurated organisms
e - Bechtel-Jacobs 1998b, 90th percentile for depurated organisms
f - Bechtel-Jacobs 1998b, 95% UPL for all organisms
g - Bechtel-Jacobs 1998b, 95% UPL for depurated organisms
h - Bechtel-Jacobs 1998b, median BSAF for all organisms
i - Fish BCFs from RAIS, User's Manual for RESRAD Version 6, ANL/EAD-4, 2001
j - Fish BCFs converted to mg/kg dry weight assuming a moisture content of 75%
Cp = concentration in plant
Cb = concentration in benthos
BAF = bioaccumulation factor, as presented by EPA (2007)
BSAF = biota-sediment accumulation factor
NA = no value available

References:
Bechtel Jacobs Company LLC. 1998a. Empirical Models for the Uptake of Inorganic Chemicals from Soil by Plants. Bechtel Jacobs Company LLC, Oak Ridge, Tennessee. BJC/OR-133.
Bechtel Jacobs Company LLC. 1998b. Biota Sediment Accumulation Factors for Invertebrates: Review and Recommendations for the Oak Ridge Reservation BJC/OR-112 Prepared for the U.S. Department of Energy Office of Environmental Management. August.
EPA. 2007. Exposure Factors and Bioaccumulation Models for Derivation of Wildlife Eco-SSLs. Attachment 4-1. Guidance for Developing Ecological Soil Screening Levels (Eco-SSLs). OSWER Directive 9285.7-55. April.

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