Quality Assurance Project Plan Addendum
Remedial Investigation
Former NIKE PR-79 Control Area
Foster, Rhode Island
DERP-FUDS D01RI0063/02

December 23, 2022

This Quality Assurance Project Plan (QAPP) Addendum provides the protocols for sample collection, handling, and storage, chain-of-custody, laboratory and field analyses, data validation, data evaluation, and reporting that are specific to soil, overburden groundwater, and bedrock groundwater water sampling and testing to be conducted at the Former NIKE PR-79 Control Area in Foster, Rhode Island ("Property"). This document summarizes updates to the Final QAPP dated September 11, 2020 for Remedial Investigation (RI); the first QAPP Addendum, dated January 2021, for residential drinking water sampling at the Property; and the second QAPP Addendum, dated May 2022, for additional RI work. RI investigations were conducted in 2020, 2021, and 2022. The field activities included herein are being conducted for the purpose of evaluating data gaps and bolstering the groundwater data set for risk assessment. Soil sampling described herein is to be considered "biased" for the purposes of filling data gaps in connection with nature and extent. This third addendum was prepared to support the United States Army Corps of Engineers (USACE) New England District with additional soil sampling, and groundwater (overburden and bedrock) sampling. Data validation protocols for soil and groundwater data will be validated to level Stage 2b. Additional soil sampling will be proposed in a future QAPP Addendum to establish an unbiased soil dataset for evaluation in the Phase II risk assessment.

Programmatic and Site-specific decisions for the RI presented in the Final QAPP as well as the first and second QAPP Addendums remain unchanged, including laboratory worksheets for methods that remain unchanged.

The following Summary of Changes for this UFP-QAPP Addendum table (Page 2) briefly outlines which worksheets have been updated from the original RI QAPP (September 2020), Residential Drinking Water QAPP Addendum (January 2021), and/or Additional RI QAPP Addendum (May 2022). The table additionally provides some information and context relative to the change included in this document in comparison to the original QAPP(s). Please refer to the September 2020 RI QAPP, January 2021 Residential Drinking Water QAPP Addendum, and May 2022 Additional RI QAPP Addendum for all other worksheets that did not require updates for the field work included herein.

Summary of Changes for this UFP QAPP Addendum

Worksheet Number and Title	Program	Matrix	Specific Change	Description/Reason for Change
9 - Project Scoping Session Participation Sheet	RI	Soil, GW, IDW	Updated per USACE and AECOM scoping discussion	Added relevant background discussions regarding QAPP scope of work
10 - Conceptual Site Model	RI	Soil, GW, IDW	Updated CSM	Updated CSM to reflect current site conditions
14 & 16 - Project Tasks and Schedule	RI	Soil, GW, IDW	Updated scope of work	Updated with additional RI scope of work
17 - Sampling Design and Rational	RI	Soil, GW, IDW	Sample rationale for additional RI field work.	Rationale updated from the original RI based on needing additional data for data gaps and risk assessment
18 - Sampling Locations and Methods	RI	Soil, GW, IDW	Sample nomenclature for additional RI sampling.	Sampling plan updated with updated sampling nomenclature and method numbers based on revised analyte list
19 & 30 - Sample Containers, Preservations, and Hold Times	RI	Soil, GW, IDW	VOCs, SVOCs, Metals (including mercury)	Worksheet is updated to include the information specific to the analyses and matrices planned for this investigation.
20 - Field Quality Control Summary	RI	Soil, GW	VOCs, SVOCs, Metals (including mercury)	Worksheet is updated to include the information specific to the analyses and matrices planned for this investigation.
20 - Field Quality Control Summary	IDW	IDW	Preliminary IDW parameters	Added estimated sample counts for potential IDW collection.
23 - Analytical Standard Operating Procedures Table	RI	Soil, GW, IDW	SOPs	SOP references were reviewed and updated to their current version for applicable methods.
24 - Analytical Instrument Calibration Table	RI	Soil, GW, IDW	VOCs, SVOCs, Metals (including mercury)	Instrument calibration information was provided for all analyses for completeness and clarity.
28 - Analytical Quality Control and Corrective Action	RI	Soil, GW, IDW	VOCs, SVOCs, Metals (including mercury)	QA/CA information was provided for all analyses for completeness and clarity.

QAPP Worksheet #9-1: Project Scoping Session Participants Sheet

Project Name: Remedial Investigation
Site Name: Former NIKE PR-79 Control Area
Projected Date(s) of Sampling: Fall 2022
Site Location: Foster, Rhode Island
Project Manager (Contractor): Kevin Kitchin
Date of Session: 14 February 2022
Location of Session: Conference Call
Scoping Session Purpose: Review of Phase 1 Risk Screening Results in Soil, Additional Biased Soil Boring Scoping Needs

Name	Title	Affiliation	Phone #	E–Mail Address	Project Role
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The following bullets summarize the significant topics that were discussed and decisions made during the review of the Phase 1 Risk Screening Results for Soil from the first phase of RI investigations and additional biased soil boring and overburden well locations. Participants included CENAE and AECOM representatives on the Project Delivery Team (PDT).

Based on PDT discussions of the 2020/2021 RI results, the team agreed to the following additional scope.

AOC-1 Radar Area

- Based on the results of soil and overburden groundwater sampling in AOC-1, in particular the detection of TCE in monitoring well MW-1, AECOM recommended the installation of two overburden/weathered bedrock wells along the northern boundary of the Property. The purpose of these wells is to clarify groundwater flow north of AOC-1 and determine if TCE is flowing to the north in overburden. The PDT agreed to the installation of these wells during the 14 February 2022 discussion.
- CENAE requested that additional geophysics be conducted along the fence line to determine the optimal placement of the two proposed overburden wells. The purpose of the additional geophysics is to identify fractures previously mapped in this area, so that the two OB wells can be installed such that they intersect one of the fractures.
- Team discussed whether or not sampling from the cess pools would be required. During the call, the Team confirmed that AMEC installed a boring in the vicinity after they found the cess pool covers, and additionally samples CessPool1 and CessPool2 were collected. Sampling results indicated VOCs were not detected. Therefore, the Team agreed that no additional borings or sampling would be needed relative to the cess pools.
- Two cess pool covers, prior "PZ" wells and any newly installed wells will be included in updated professional survey.
- The PDT discussed the cumulative RI soil results from AOC-1 and ultimately agreed that no additional biased soil samples would be required, however biased soil samples were requested in the July 2022 PWS pertaining to this QAPP Addendum. Only unbiased surface soil samples collected via Incremental Sampling Methodology (ISM) would be needed to establish the soil dataset for the Phase II risk assessment to be conducted in the RI following completion of the RI field investigations. The Team discussed potentially dividing the AOC-1 Decision Unit (DU) into two smaller (half-acre) Sampling Units (SUs), north and south. Ms. Lapite suggested considering three SUs in order to accommodate the need for triplicate QC sampling. (Note that sampling design for unbiased/ISM sampling needs will be included in a future QAPP Addendum).

- Ms. Kirby stated that arsenic detections may be due the historic use of the area as an orchard (pesticides). Ms. Ouellette stated that arsenic may also be naturally occurring.

AOC-2 Operations and Maintenance Area

- The PDT discussed the cumulative RI soil results from AOC-2 and ultimately agreed that no additional biased soil samples would be required. Only surface soil samples collected via ISM would be needed. The Team discussed potentially dividing the AOC-2 DU into two to three smaller SUs, north and south, with the southern SU potentially divided further around soil borings SB-012 and SB-013. (Note that sampling design for unbiased/ISM sampling needs will be included in a future QAPP Addendum).

AOC-3 Southern Leach Field

- The PDT discussed the cumulative RI soil results from AOC-3. Prior to determining if unbiased/ISM sampling will be needed at AOC-3, the PDT ultimately agreed to the collection of additional biased soil sampling and overburden wells to further characterize:
    - 1) The source of an elevated detection of benzo(a)pyrene in boring SB-113 located north of the Former Mess Hall. Potential sources include the grease intercept and the former heating oil UST (both located adjacent to the north side of the building), and the former septic system/leach field (located west/southwest of the building).
    - 2) The source of TCE detected at the seep at sampling location WT007, located southwest of AOC-3. Potential sources include the septic system and the associated clay discharge pipe that flows from the septic to the downgradient sewage disposal area to the west; and TCE-impacted groundwater from the Site.
- The PDT agreed to the installation of three biased soil borings in AOC-3:
    - One soil boring between the Former Mess Hall (where historic boring SB-113 was located) and the septic tank (where historic boring SB-112 was located) (soil sampling only);
    - One soil boring between the septic tank (where former boring SB-112 was located) and the property boundary (to be completed as an overburden/weathered bedrock monitoring well as a replacement boring to the original SB-112); and
    - One soil boring along the clay pipe north of the seep area (to be completed as an overburden/weathered bedrock monitoring well).
- If it is ultimately determined that the benzo(a)pyrene is related to the leaking UST, additional soil sampling by USACE would not be warranted.

- The Team discussed extending the boundary of AOC-3 to include wells MW-006 and MW-007.

AOC-4 Western Sewage Disposal Area

- The PDT discussed the cumulative RI soil results from AOC-4. The Phase I screening results indicated arsenic was the only chemical detected in soil at a concentration above human health screening levels and background threshold values (BTVs) (i.e., 95 percent upper tolerance limit within the Site-specific background soil dataset) (in two soil borings, SB-117 and SB-026). No chemicals of potential ecological concern were identified in AOC-4 based on the Phase I screening results. Arsenic was not identified as a chemical used at NIKE missile batteries and is unlikely to be related to former Department of Defense (DoD) activities (USACE EMCX, 2003). Therefore, no further soil sampling was recommended in the vicinity of AOC-4 relative to the RI Risk Assessment and the team ultimately agreed that no additional soil samples (biased or ISM) would be required.

Subsequent to the 14 February 2022 PDT discussion, CENAE requested in an email dated 20 April 2022 the addition of a bedrock monitoring well, to be located south of the Property, between the Property and sample location DW-39 (located at 39 Windsor Road), where TCE has been detected periodically.

QAPP Worksheet #10: Conceptual Site Model

10.1 Overview

This worksheet presents general background information and the general updated Conceptual Site Model (CSM) for the Site. A more detailed CSM was provided in the Final QAPP dated September 11, 2020. This updated CSM is subject to change based on the results of this investigation. A more detailed updated CSM will be included in the RI Report.

10.2 Conceptual Site Model Summary

The following subsections briefly summarize the current CSM. It is divided into the specific AOCs identified for investigation, shown in Figure 10-1. Sample locations are depicted on Figures 10.2 and 17-1A and 17-1B.

AOC-1 Radar Area

Study Area: The study area includes a former helicopter pad and former target acquisition and tracking radar equipment located at Radar Pad A, Radar Pad B, Radar Pad C, Radar

Control Van, Battery Control Van, Frequency Changer/Generator building (including a floor drain), associated cesspools and a dry well.

The floor drain was previously investigated in the initial phase of the RI. Upon uncovering the floor drain and associated drainage trench. The initial phase of the RI found VOCs (including TCE at an estimated low concentration [0.0009 J mg/L]) in a groundwater. The 2020-2021 RI evaluated VOCs in the underlying overburden and weathered bedrock zones.

Dissolved TCE was identified above Project Action Limits (PALs) during the 2020 RI in groundwater samples collected from newly installed overburden well PR79-MW-003, suggesting that AOC-1 (the former Frequency Changer Generator Building) is a potential source area of TCE. AOC-1 is located near the topographic high point of the Property. Overburden groundwater elevation contours indicate that overburden groundwater flow may be radial away from this topographical high point; however, there are currently no overburden monitoring wells to the north and northeast to confirm this interpretation.

The primary migratory pathway of dissolved TCE in groundwater appears to be from the overburden material in the AOC-1 source area to the underlying bedrock and through the major transmissive fracture zones identified to the south and southwest. Dissolved TCE has been confirmed in bedrock wells in those directions (i.e., wells PR79-BR-002, PR79-BR-001, PR79-BR-003, NIKE-1, ROU-1, ROU-2, ROU-3, and DW-39) and bound by non-detection results in wells DW-41 and DW-37. Bedrock well PR79-BR-002 is the northernmost well on the property. This well has some of the highest impacts in fractures that dip to the north/northeast with elevated head. Although there has been no indication of TCE in drinking water wells to the north (DW-68 and DW-69), northeast (DW-24) and east (DW-21).

AOC-2 Operations and Maintenance Area

Study Area: The study area includes the former Mess Hall, Barracks, and Administration buildings, water pump house, former transformer area, and reported motor pool vehicle maintenance areas located northwest of the former Guard Post along Theodore Foster Drive and northwest of the former water storage tank and pump house. The study area also includes an “unidentified pipe” observed west of former water pump house which is approximately 23 feet deep and is thought to be an abandoned borehole.

The transformers and associated electrical equipment were removed by the Town of Foster after being transferred to the Town of Foster in 1965 for beneficial use. The former transformer area was previously investigated twice, and the results from surficial soil samples indicated PCBs were not present at concentrations exceeding laboratory reporting limits.

Based on the findings of these previous investigations, and that the electrical equipment was beneficially used and removed by the Town of Foster, further investigation of the former transformer area was not conducted as part of the 2020-2021 RI.

Soils in the vicinity of the Former Vehicle Maintenance area have indicated concentrations of benzo(a)pyrene and lead at or above their respective PALs in samples collected at PR79-SB-109. Based on the 2020-2021 RI, both benzo(a)pyrene and lead appear to be delineated both vertically and horizontally, isolated to boring PR79-SB-109.

Potential impacts from TCE and other degreasing solvents are related to the maintenance of former buildings and potentially used for motor pool vehicle maintenance. Gasoline, diesel, and motor oil leaks and spills related to former motor pool vehicle maintenance was investigated as part of the 2020-2021 RI with the installation of boring/overburden monitoring well PR79-SB-109/PR79-MW-005 and bedrock monitoring well BR-004. TCE was not reported at concentrations exceeding laboratory detection limits in groundwater samples collected at PR79-MW-005 and/or PR79-BR-004.

The absence of TCE in groundwater and horizontal and vertical delineation of soils in the vicinity of the Former Barracks and Former Vehicle Maintenance area my indicate that these features in AOC-2 are not to be considered potential source areas. As a result, no further investigation is warranted at this time.

AOC-3 Southern Leach Field

Study Area: The study area includes the former southern leach field, former distribution box, and drain line connected to the Western Sewage Disposal Area.

Potential impacts of TCE and other degreasing solvents used as cleaning agents and paint waste disposed through the septic system were investigated during the 2020-2021 RI. Monitoring wells PR79-MW-006 and PR79-MW-007 were installed to target the shallow water bearing unit adjacent to the former Mess Hall for human health risk assessment and assess potential overburden impacts from the former leach field and septic tank, respectively.

Benzo(a)pyrene weas detected at a concentration above its respective PAL in samples collected at boring PR79-SB-113. Benzo(a)pyrene appears to be vertically delineated. Elevated concentrations of benzo(a)pyrene in subsurface soil at PR79-SB-113 may be related to a leaking 2,000-gallon No. 2 heating oil underground storage tank (UST) located approximately 50 ft east of PR79-SB-113. Further evaluation of soils in this area was determined to be warranted.

Nitrite was detected at an estimated concentration (210 µg/L) slightly above the associated PAL, equal to the USEPA Regional Screening Level (RSL) for tapwater based on a target hazard quotient of 0.1 (May 2022), of 200 µg/L in a groundwater sample collected from PR79-MW-006. The estimated nitrite concentration of 210 µg/L in groundwater is less than the USEPA Maximum Contaminant Level (MCL) of 1,000 µg/L. Nitrite was not detected in the background bedrock groundwater well (i.e., Steere well). PR79-MW-006 is located approximately 100 feet from an active septic leach field constructed in the 2000s by the Town of Foster to replace a former septic system; therefore, nitrite is likely associated with that septic leach field. No other analytes were identified at concentrations exceeding laboratory detection limits in groundwater samples collected from PR79-MW-006 and/or PR79-MW-007.

Three heating oil USTs that supplied the Barracks, Administrative, and Mess Hall buildings were transferred to the Town of Foster in 1965 On December 16, 2019. To date, the three USTs have been removed. Upon removal, indication of a release was observed from the UST located adjacent to the former Mess Hall. The Formerly Used Defense Site (FUDS) Program Policy, ER 200-3-1, provides specific criteria for property eligibility. The Program Policy expressly provides that USTs that have been beneficially reused by the Property Owner are ineligible under the FUDS Program.

Currently, RIDEM is requiring the Town of Foster to conduct quarterly groundwater sampling of up to eight shallow overburden monitoring wells installed by Sage Environmental. Groundwater monitoring of SVOCs is to occur during the months of January, April, July, and October of each year until further notice.

AOC-4 Western Sewage Disposal Area

Study Area: The study area includes the former sand filtration beds, former chlorine detection chamber and former utility shed located at the Western Sewage Disposal Area.

The location of the utility shed in the Western Sewage Disposal Area was investigated in the initial phase of the RI (AMEC, 2013) with soil borings SB-023 and SB-026. Additional soil sample locations in this AOC were sampled as part of the 2020-2021 RI (PR79-SB-117, -118, and -119). Compounds were not identified at AOC-4 at concentrations exceeding screening levels, except for arsenic, which was reported in 2 of 5 surface soil samples (at a maximum concentration of 3.29 mg/kg) and 1 of 3 subsurface soil samples (at a maximum concentration of 2.61 mg/kg) at concentrations exceeding the May 2022 Residential Soil Regional Screening Level (RSL) of 0.68 mg/kg and the Site-specific BTVs of 2.6 mg/kg and 1.7 mg/kg for surface soil and subsurface soil, respectively. Arsenic is commonly found in soils in Rhode Island and has not been identified as a chemical used at NIKE missile batteries and is unlikely to be

related to former DoD activities (USACE EMCX, 2003). Furthermore, elevated concentrations of arsenic were not identified in borings placed in areas leading into (PR79-SB-116 and -115, collected from 5-6 and 5-7 feet, respectively) or out of (PR-79-SB-119, collected from the surface down to 8-10 feet) the former sand pit area. This further supports that the arsenic identified in this area is likely a background condition.

As part of the 2020-2021 RI soil boring/monitoring well PR79-SB-118/PR79-MW-008 was installed at the downgradient edge of the former Sand Pits to assess whether they affected groundwater quality. Numerous attempts have been made to sample monitoring well PR79-MW-008; however, the well has been dry (i.e., no water column) since its initial installation.

The absence of COPCs in soil samples collected as part of the 2020-2021 RI suggests the area surrounding the former sand filtration beds is not a source for impacts to soil. Attempts will continue to be made to monitor groundwater conditions in well PR79-MW-008. No further investigation of AOC-4 is warranted at this time.

AOC-5 Western Disposal Area

AOC-5 was used for residential and agricultural dumping by the property owner and is not attributable to former DoD activities; therefore, AOC-5 is ineligible for investigation and cleanup under DERP-FUDS.

10.3 Data Gap Analysis

The review of the historic information and working CSM has identified data gaps in the technical understanding of the hydrogeology, migration pathways, and nature and extent of impacts. A detailed data gap analysis was included in the September 2020 RI QAPP. The following briefly outlines data gaps developed following RI activities conducted from 2020 to 2021. Sample locations discussed herein are depicted on Figure 10.2.

Hydrogeology and Migration Pathways

Groundwater flow: Overburden groundwater contours indicate that overburden groundwater flow may be radial away from this topographical high point; however, there are currently no overburden monitoring wells to the north and northeast that would confirm this interpretation. The installation of two additional overburden monitoring wells to better define the extent and potential migration of TCE in overburden groundwater from the probable source area around PR79-MW-003 to the north and northeast. Overburden wells should be screened in the same lithologic units as MW-003 (i.e., into weathered bedrock, assumed depth of wells to be approximately 35 feet).

The primary migratory pathway of dissolved TCE in groundwater appears to be from the overburden material at the AOC-1 source area (former Frequency Changer/Generator Building) to the underlying bedrock and through the major transmissive fracture zones identified to the south and southwest. Dissolved TCE has been confirmed in bedrock wells in those directions (PR79-BR-002, PR79-BR-001, PR79-BR-003, NIKE-1, ROU-1, ROU-2, ROU-3, and DW-39) and bound by non-detection in wells DW-41 and DW-37. Bedrock well PR79-BR-002 is the northernmost well on the property. This well has some of the highest impacts in fractures that dip to the north/northeast with elevated head. Although there has been no indication of TCE in drinking water wells to the north (DW-68 and DW-69), northeast (DW-24) and east (DW-21).

Downhole bedrock fracture characterization: An additional bedrock well is needed to intercept and further characterize potential migration pathways via transmissive fractures and measure the vertical component of the hydraulic gradient within bedrock to the south/southwest of residential wells ROU-2 and ROU-3.

Nature and Extent of Impacts in Soil, Overburden, and Bedrock

Plume delineation (horizontal and vertical): The existing dataset for soil and groundwater is not sufficient to adequately assess the horizontal and vertical extent of impacts, particularly in deep overburden, weathered bedrock, and bedrock. The current data is limited with respect to the media assessed and the likely pathways in which COPCs migrated over time. Previous investigations concluded that TCE, pentachlorophenol, and naphthalene are the only Site-related COPCs identified in overburden groundwater and are isolated to the area surrounding PZ-019 located immediately adjacent to the former Frequency Changer/Generator Building as well as in the area surrounding PR70-MW-003 in the Radar Area (AOC-1). Additional data is needed to assess this potential source area in overburden, identify whether a discrete or diffuse continuing source exists, and assess transport mechanisms in weathered bedrock and bedrock. This will be achieved by installing new monitoring wells screened to target permeable overburden and transmissive bedrock fractures and conducting sampling for COPCs and geochemical parameters to understand the chemical and physical processes impacting the plume.

10.4 Description and Current Use

The Former NIKE PR-79 Control Area (Property) is located in Providence County, in Foster, Rhode Island, as shown on Figure 10-1. The coordinates for the Site obtained from the US Geological Survey (USGS) 7.5-minute Quadrangle for Clayville, Rhode Island are approximately:

- Latitude: N41° 50' 32"
- Longitude: W71° 42’ 57"

The Property is located at the end of Theodore Foster Drive. According to the Town of Foster Tax Assessor Online database, the address is 23 Theodore Foster Road and recorded on Lot 10, Map 18. The parcel is 6.62 acres in size[1]. The land is zoned for municipal use.

- The area surrounding the Former NIKE PR-79 Control Area is comprised of northern hardwood forest and rural development. The Property is located on top of Oak Hill and residences, farms, and businesses are located south of the Property. Three residences are located within 300 to 400 ft of the Property with approximately 68 residences located within a one-mile radius with private water supply wells in Foster and North Scituate, Rhode Island. The majority of these homes are located along Maple Rock Road, Winsor Road, and Old Hartford Pike. The nearest residential private water supply well (ROU-1) is located approximately 200 ft east of the Property. Businesses located near the Property include solar panel arrays located to the northeast and southeast of the Property. The location of on-Property supply wells (NIKE-1, NIKE-2), nearby residential water wells (ROU-1, ROU-2, and ROU-3) and solar panel arrays are shown on Figure 10-2.

The Town of Foster has recently converted the former Mess Hall building to be used for residential uses. There is currently one tenant occupying the former Mess Hall building. The former mess hall building, which is the only building on the Former NIKE PR-79 Control Area property, was temporarily declared "unsafe" in July 2020 by the Town of Foster Building Inspector due to potential asbestos containing building materials.

10.5 Topography and Geology

The Former NIKE PR-79 Control Area is located on top of Oak Hill at approximately 620 ft above mean sea level (msl). The surrounding terrain is characterized by low hills and shallow valleys. The elevation of Winsor Brook is approximately 470 ft above msl.

The surficial geology in the vicinity of the Site is made up of glacial-fluvial deposits in the valleys and lodgment and ablation till along the hills. The glacial outwash is composed of sand and gravel interbedded with silt and clay. The units form unconsolidated and generally well sorted and

1 Town of Foster Tax Assessor Online database indicates the parcel is 6.62 acres in size; however, there is conflicting information from other sources regarding the exact acreage. The DERP-FUDS Inventory Project Report indicates the Site is 19.59 acres in size (13.36 acres and a 6.23-acre easement). Historic reports from Camp Dresser & McKee, Inc. (CDM) and CENAE reference 8 acres (CDM, 1994; CENAE, 2003). On September 22, 1995, 1.38 acres was transferred to Lot 11 on Plat 18 (AMEC, 2013).

stratified sequences that reach up to 50 ft thick in the area surrounding the Site. Site-specific geophysical and boring log information indicates a much thinner layer of overburden of between 5 to 35 ft thick. Boulders, sand, silt, and clay are found within the poorly sorted and unstratified glacial materials. As observed during the 2020 RI field events, glacial till forms a thin, discontinuous mantle over the bedrock surface averaging 18 ft in thickness.

Below the overburden material is a layer of weathered bedrock. Surface geophysics performed at the Site indicate the thickness of this layer varies between one and 15 ft beneath the Site. However, the weathered bedrock is sometimes thicker and the bedrock surface elevation lower in areas of highly fractured bedrock. The bedrock beneath the Site is composed of the South Foster Migmatite and Ponaganset Gneiss of the Esmond Igneous Suite. The South Foster Migmatite consists of a heterogeneous composite of quartz-biotite schist and quartzite members. The Ponaganset Gneiss is a coarse grained, porphyritic, pink to gray diorite gneiss. The gneiss is the most predominant rock type across the Site.

Based on borehole geophysics and outcrop field observations, the competent bedrock is characterized by steeply dipping fractures and occasional fracture zones spaced from approximately 4 to greater than 10 ft apart. The orientations of field-measured bedrock fractures are similar to the orientations of topographic lows feeding into Barden Reservoir located south of the Site, suggesting that ground surface topography is sometimes controlled by preferential erosion along zones of closely spaced bedrock fractures.

The steeply dipping fracture orientations in bedrock predominantly strike north-northwest (NNW) to south-southeast (SSE) and north-northeast (NNE) to south-southwest (SSW) on Site, although north to south and east-northeast (ENE) to west-southwest (WSW)-striking fractures are also present. The primary water-bearing fracture sets encountered in Site wells NIKE-1, NIKE-2, ROU-1, ROU-2, and ROU-3 strike NNE to SSW and NW to SE and dip moderately.

South of the Site, NNW to SSE and northwest (NW) to southeast (SE)-striking fractures are more prevalent, reflecting the primary orientation of the Ponaganset River drainage to Barden Reservoir. These fracture strike orientations are reflected in the topographic drainage patterns, suggesting that the bedrock joints and associated fracture sets represent preferential erosional zones.

10.6 Hydrology and Hydrogeology

There are no surface water bodies in the Former NIKE PR-79 Control Area (Site). Surface water runoff from the Site is directed into drainage ditches which flow along the slopes of Oak Hill. Surface water not captured by these ditches infiltrates or flows radially in all directions, since the

Former NIKE PR-79 Control Area is located on top of a hill. The nearest surface water bodies include three streams and a 16-acre wetland complex located approximately 0.25 mile to the south; a 0.15-acre wetland followed by Winsor Brook located approximately 0.25 miles to the west; and a 0.07-acre open water body to the north of the Site. The Site is located within the Scituate Reservoir Watershed and is within the Scituate Reservoir Protection Area. The northwestern most portion of the Scituate Reservoir (known as the Barden Reservoir) is located approximately 3 miles southeast of the Site. Winsor Brook is a tributary to the Ponaganset River, which flows into the Barden Reservoir. Local potable water is supplied with private bedrock drinking water supply wells, not municipal water. There are no wellhead protection areas within one mile of the Site. The groundwater beneath the Site is classified as Group GA which is presumed to be suitable for drinking water.

The main source of overburden groundwater at the Site comes from infiltration. Precipitation and melt water infiltrate and likely form a saturated zone in the coarse-grained sand and gravel above the less permeable till and bedrock. Over time, the overburden groundwater slowly drains both horizontally and vertically into the weathered bedrock and competent bedrock fractures below or migrates radially from the top of the hill likely contributing to seeps and wetlands observed at the base of Oak Hill. Groundwater within bedrock likely receives water from a series of fractures which are recharged from the overlying glacial overburden.

Surface geophysics, fracture trace analysis, and borehole geophysics investigations have been completed at the Site. The integration of these data indicates that fracture strikes trend primarily north to south and dip steeply to the southwest and southeast. Large low-velocity anomalies indicating fracture zones at the Site were identified along seismic and GPR geophysical lines. The orientation of fractures observed at outcrops and identified in fracture domain analysis provide evidence that a fracture zone could connect supply wells NIKE-1 and ROU-1. Borehole geophysical logging at supply wells NIKE-1, NIKE-2, ROU-1, ROU-2, ROU-3, and bedrock monitoring wells PR79-BR-001 through PR79-BR-005 identified 25 water producing fracture sets dipping to the southwest and southeast that intersect two or more supply wells. The deepest three fracture sets were identified as primary water producing fractures. During 2013, AMEC performed a four-hour pump test in NIKE-2 to assess hydraulic connectivity between NIKE-1, ROU-1, ROU-2, and ROU-3. AMEC concluded that NIKE-1 and ROU-1 are hydraulically connected and there is evidence of weak influence and connectivity between NIKE-1 and NIKE-2.

Groundwater flow from the Site has a radial character, reflecting the elevated topography of the Site with components of flow ranging from the west to southeast. There is an ENE drainage from Oak Hill, as well, but that is located over 1,000 feet northeast of the previously active portions of the Site and therefore likely has less potential for influencing contaminant transport. The

predominant topographic drainage patterns in near-Site area are generally SSW to the west of the Site and SSE to the south and east of the Site. The westerly and south-southeasterly drainage features converge south of the Site and converge with the Ponaganset River in close proximity to one another approximately 0.7 miles SSW of the Site.

10.7 Operational History and Environmental Areas of Concern

The Property was originally developed for agricultural use, namely as an apple and peach orchard. The US Government acquired the subject property between 1955 and 1957 and developed it for radar missile tracking as part of the NIKE Missile Defense System. NIKE sites were constructed throughout the continental US in the mid-1950s during the Cold War era to defend major industrial and urban areas. The location of NIKE PR-79 was selected for defense of Providence, Rhode Island. NIKE sites generally consisted of a missile launcher area and a separate integrated fire control and radar missile tracking area (NIKE control area) which typically operated less than two miles apart. The launcher area is where missiles were stored, maintained, and if necessary, launched. The NIKE control area is where radar and communication equipment needed to detect potential targets and guide launched missiles were maintained and stored. The former launcher area for NIKE PR-79 is a separate property located on Winsor Road in Foster, Rhode Island designated FUDS Property/Site Number D01RI0063/01 and is not the subject of this document.

The Former NIKE PR-79 Control Area was reported as excess property by the General Services Administration (GSA) in 1964. In July 1965, the Site was closed and the Property was transferred to the Town of Foster. The Town of Foster used the former Mess Hall, Barracks, and administrative buildings as the Fogarty Elementary School until 1989 (RIDEM, 1992). The Town of Foster recently retrofitted the Site building to utilized as residential. A residential tenant currently occupies the former Mess Hall building.

The following structures were transferred to the Town of Foster in good condition for beneficial reuse:

- Mess Hall, Barracks, and Administrative buildings
- Heating Oil USTs supplying the Mess Hall, Barracks, and Administrative buildings
- Utility Lines
- Southern Leach Field

The locations of the above referenced structures are shown on Figure 17-1B.

In 1988, the Foster Board of Education requested that CENAE investigate groundwater at the Former NIKE PR-79 Control Area to determine whether TCE detected by RIDEM in water supply

wells was related to former DoD activities. CENAE conducted a field survey and Inventory Project Report (INPR) that same year, which concluded that former DoD activities may have resulted in the release of TCE to the environment. Based on the findings of the INPR, the Former NIKE PR-79 Control Area entered DERP and was designated FUDS Property/Site Number D01RI0063/02 (CENAE, 1988).

The INPR field survey identified a 6,000-gallon diesel fuel UST (originally assumed to be a 1,000-gallon UST) in the northeast corner of the Former NIKE PR-79 Control Area, next to the former Frequency Changer/Generator Building shown in Figure 10-1. The UST was removed in June 1994. There is limited documentation beyond that the UST was closed under RIDEM UST regulations and no additional UST investigation was recommended.

In March 1992, USEPA designated the Former NIKE PR-79 Control Area as Site Number RID987492485 in Comprehensive Environmental Response, Compensation and Liability Information System (CERCLIS). CERCLIS is a management system used by the USEPA to track activities at hazardous waste sites considered for cleanup under CERCLA. The Site is not included on the National Priorities List (NPL).

On August 24, 2000, RIDEM issued a Letter of Responsibility (LOR) to the US Army 94th Regional Support Command at Fort Devens, Massachusetts, indicating that a potential release of hazardous materials occurred at the Site and identified DoD as the potentially responsible party. The LOR requested the US Army conduct a site investigation of the source area in accordance with Rhode Island Remediation Regulations 250-RICR-140-30-1. On September 5, 2000, the US Army 94th Regional Support Command sent a response letter to RIDEM refuting ownership of the Site.

Beginning in 2001, CENAE conducted a series of residential water supply well sampling events, which targeted two on-Property water supply wells (NIKE-1, NIKE-2) and three off-Property residential water supply wells located at 23A Theodore Foster Drive (ROU-1, ROU-2 and ROU-3) that are in close proximity to the Former NIKE PR-79 Control Area. Based on the analytical results for the residential sampling, in 2002, a Time Critical Removal Action (TCRA) was initiated as a temporary remedy for groundwater impacts in drinking water. The TCRA included installation of point of use dual carbon filtration systems at one on-Property water supply well (NIKE-1) and three off-Property residential water supply wells (ROU-1, ROU-2, and ROU-3). CENAE continues to monitor these four carbon filtration systems.

Five AOCs have been identified based on previous investigations:

- AOC-1: Radar Area
- AOC-2: O&M Area

- AOC-3: Southern Leach Field
- AOC-4: Western Sewage Disposal Area
- AOC-5: Western Disposal Area (ineligible for investigation and cleanup under DERP-FUDS)

Based on a review of historic aerial photographic analysis, historical maps, and other documents regarding historical operations, there is no evidence to suggest fire training activities or other activities using per- and polyfluoroalkyl substances (PFAS) occurred at the Former NIKE PR-79 Control Area. Additionally, DoD activities at the Former NIKE PR-79 Control Area precedes dates of common PFAS use as aqueous film forming foam (AFFF). Therefore, PFAS is not a Site-specific COPC.

10.8 Previous Site Investigations and Available Dataset

A detailed discussion of previous investigations at the Former NIKE PR-79 Control Area including a Preliminary Assessment (PA) and Site Inspection (SI), off-Property residential and on-Property water supply well sampling, soil, groundwater, and soil vapor studies, and surface and geophysical investigations was provided in the September 2020 RI QAPP. Site investigation activities that occurred as part of recent RI activities between 2020 and 2021 are summarized below.

2020-2021 Remedial Investigation

RI activities conducted were presented in the Draft RI Field Report dated April 22, 2021 and revised August 19, 2021. The Field Report presented the data collected during RI field activities occurring between April 2020 and February 2021. RI field activities were conducted in accordance with the QAPP (original final dated April 10, 2020 and updated September 11, 2020) and consisted of the following:

- Background Soil Sampling;
- Surficial and Subsurface Soil Sampling;
- Monitoring Well Installation and Development;
- Hydraulic Conductivity Testing;
- Piezometer Abandonment;
- Borehole Geophysical Testing;
- Flexible Liner Underground Technologies, LLC (FLUTe) Activated Carbon Technique (FACT) Testing;
- Bedrock Fracture Connection Testing;
- Water FLUTe Multi-Level Sampler (MLS) Installation and Development;
- Background Groundwater Sampling;

- Groundwater Sampling;
- Background Surface Water, Porewater, and Sediment Sampling;
- Surface Water, Porewater, and Sediment Sampling;
- Water Supply Sampling;
- Land Surveying;
- Laboratory Data Validation; and
- Data Evaluation.

Analytical results were compared to PALs, and a Phase I risk screening assessment was performed in accordance with Section 2 of the risk assessment work plan (included as Appendix G of the QAPP) to identify additional data gaps and sampling needs to support completion of the RI Report.

Based on the 2020 RI phase field activity findings, the following data gaps were identified:

- Several monitoring wells were dry when water levels were measured in Summer 2020 and Fall 2020. To further evaluate seasonal fluctuation in water levels and potential for migration through overburden materials, collection of synchronous water level measurements seasonally was recommended.
- Overburden monitoring well PR79-MW-008 was dry when groundwater sampling was attempted in Fall 2020 and Winter 2021. PR79-MW-008 is currently the only overburden monitoring well located in AOC-4 to evaluate groundwater conditions in that AOC. Another groundwater sampling attempt at PR79-MW-008 was recommended during a period of elevated water elevations. If a groundwater sample cannot be collected, then alternatively, a surface water and porewater sample was recommended at the base of the slope immediately west of PR79-MW-008 to assess water quality emanating from AOC-4.
- Confirmatory sampling of the five existing piezometers, eight overburden wells that were installed during the RI, and five bedrock wells that were installed during the RI was recommended to supplement the RI risk assessment data set.
- The RI Report will further evaluate the saturated thickness of overburden, stratigraphy, and hydrogeology of the hilltop to determine the extent and possible migration pathways for TCE detected in overburden groundwater at AOC-1. Seasonal water level information will support this evaluation. Additional exploratory soil borings may be necessary to better define the extent and potential migration of TCE in overburden groundwater and into bedrock from the probable source area around PR79-MW-003.
- The RI Report will further evaluate in more detail potential transport pathways in transmissive fracture zones for TCE from the probable source area around PR79-BR-002 to NIKE-1, ROU-1, ROU-2, and ROU-3.

- Additional soil, sediment, porewater and surface water sampling was required to supplement the RI risk assessment and background evaluation.

Supplemental sampling of residential wells, overburden groundwater wells, bedrock groundwater wells, and surface water/pore water locations has been completed through June 2022. Results of this sampling will be described in a future Field Report deliverable. Additional soil sampling will be proposed in a future QAPP Addendum to establish an unbiased soil dataset for evaluation in the Phase II risk assessment.

10.9 Receptors and Exposure Pathways

The human health and ecological receptors and potentially complete exposure pathways under current and reasonably anticipated future land use scenarios to be considered for the Site are summarized below. The Former NIKE PR-79 Control Area FUDS property is currently zoned as "municipal". However, the Town of Foster recently retrofitted the current on-Site building (former Mess Hall building) for residential use. The area to the south of the FUDS property is currently residential use. The DoD is implementing a Time Critical Removal Action (TRCA) to treat residential drinking water wells, in which Site-related VOCs have been identified, in this off-Property residential area. Future use of the FUDS property and the surrounding area is anticipated to remain consistent with current use. However, recreational use of the FUDS property is also considered a reasonable future use scenario. An unlimited use and unrestricted exposure (UU/UE) scenario will be evaluated to provide information for making risk-management decisions.

Human Health

Current/future human receptors at the Site are as follows:

- Current/Future On-Property Resident (Adult/Child)
- Current/Future On-Property Trespasser (Adolescent);
- Current/Future On-Property Commercial/Industrial Worker;
- Current/Future On-Property Construction/Utility Worker;
- Current/Future Off-Property Resident (Adult/Child); and
- Future Recreational User (Adult/Child).

The potentially complete exposure scenarios for the above receptors and Site media are as follows:

- Exposure to surface soil may occur by current/future on-Property residents, trespassers, and commercial/industrial workers, and future recreational users. Surface

soil exposure pathways include incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles.

- Exposure to the combined surface and subsurface soil interval (to a maximum depth of 10 ft) may occur by current/future on-Property construction/utility workers and by future on-Property residents, trespassers, commercial/industrial workers, and recreational users, assuming soils become mixed during potential future redevelopment activities. Soil exposure pathways include incidental ingestion, dermal contact, and inhalation of particulates and/or volatiles.
- Exposure to shallow groundwater may occur by:
    - current/future on-Property construction/utility workers via incidental ingestion, dermal contact, and inhalation of volatiles in an excavation trench.
    - current/future on-Property residents and commercial/industrial workers via inhalation of indoor air within the current on-Property building and hypothetical on-Property buildings that may be constructed in the future (i.e., potential vapor intrusion pathway).
    - current off-Property residents via inhalation of volatiles in indoor air (i.e., potential vapor intrusion pathway).
- Exposure to groundwater (from on-Property monitoring wells [overburden and bedrock], piezometers, and water supply wells [using groundwater collected prior to carbon filtration]) may occur by:
    - future on-Property commercial/industrial workers via ingestion of drinking water. (This exposure pathway is not complete under a current scenario due to the presence of a carbon filtration system on existing on-Property water supply well NIKE-1 and NIKE-2 is inactive. Evaluation of the future scenario will also provide information on a hypothetical scenario in which the carbon filtration system on NIKE-1 fails)
    - current/future on- and off-Property residents via ingestion of drinking water, dermal contact and inhalation while bathing/showering. (This exposure pathway is not complete under a current scenario due to the presence of a carbon filtration system on existing water supply wells in closest proximity to the Property. Off-Property residential water supply wells located further away from the Property ("DW-wells") do not all have carbon filtration systems installed. These wells will not be evaluated within the scope of the risk assessment. However,

recommendations for further assessment of these wells during a later RI phase may be made following assessment of groundwater results from the on-Property area and adjacent off-Property area.)

- Exposure to sediment and surface water in water bodies potentially impacted by Site groundwater may occur by future recreational users via incidental ingestion (sediment only) and dermal contact (sediment and surface water) while wading.

Ecological

The undeveloped, wooded portions of the Site are expected to provide habitat for ecological receptors such as plants, soil invertebrates, small birds and mammals, and reptiles and amphibians. These receptors may be directly exposed to Site-related constituents released to the surface soil (e.g., terrestrial plants, earthworms) or via ingestion of impacted food items (e.g., birds or mammals consuming impacted earthworms).

Ecological receptors are typically not directly exposed to groundwater. However, exposure to constituents present in groundwater may occur when groundwater discharges into a water body such as Winsor Brook, or contributes to seeps and the forested swamp located to the south of the Property. Ecological receptors in the brook, the seeps, and the forested wetlands may be exposed to Site-related groundwater constituents as groundwater is discharged into porewater, sediment, and eventually surface water in these areas. Aquatic receptors such as invertebrates, fish, or amphibians may be directly exposed to constituents in the water column and benthic (sediment-dwelling) invertebrates may be directly exposed to constituents in the sediment or porewater (as groundwater discharges through the sediment into the water body). Birds and mammals may be exposed to constituents in water bodies through the incidental ingestion of sediment, ingestion of drinking water, or ingestion of impacted food items.

Therefore, the ecological exposure scenarios most likely to be complete at the Site include the following:

- Direct contact with surface soil by terrestrial plants and invertebrates;
- Incidental ingestion of surface soil and ingestion of impacted food items by terrestrial birds and mammals in terrestrial areas which provide habitat for wildlife;
- Direct contact with sediment by benthic/wetland invertebrates in water bodies potentially impacted by Site groundwater;
- Direct contact with pore water (i.e., Site groundwater discharging into water bodies) by benthic invertebrates;

- Direct contact of surface water by amphibians, aquatic invertebrates and/or fish in water bodies potentially impacted by Site groundwater; and,
- Ingestion of sediment, surface water, and impacted food items by semi-aquatic birds and mammals foraging in water bodies potentially impacted by Site groundwater.

QAPP Worksheets #14 /16: Project Tasks & Schedule

The following project tasks will be performed as part of the Addendum (refer to WS18 and 20 for specific VOC and SVOC method for each matrix as bottleware, preservation, etc. may differ):

- Advancement of two (2) soil borings north of AOC-1 along the property boundary (to be completed as overburden/bedrock interface wells)
- Advancement of one (1) soil boring between the former heating oil UST and septic tank at AOC-3
- Advancement of one (1) soil boring north of the seep near porewater sample WT-007. Boring will be placed south of the clay drainage pipe that runs from the former septic tank at AOC-3 to AOC-4 (to be completed as an overburden/bedrock interface well)
- Advancement of one (1) replacement soil boring, formerly SB112, that will be moved away from the existing septic tank to a location between the septic tank and the property boundary at AOC-3 (to be completed as a well).
- Advancement of one (1) soil boring to be completed as an interceptor bedrock well downgradient of the FUDS and drinking water wells ROU2 and ROU3, but upgradient of the Winsor Road residences.
- Collect one (1) surficial and one (1) subsurface sample from each of the soil borings. The soil samples will be submitted for analysis of VOCs (full scan), SVOCs SIM (including 1,4-dioxane and pentachlorophenol), and total metals.
- Two (2) rounds of overburden groundwater monitoring well sampling at four (4) newly installed monitoring wells, for VOCs (full scan and SIM), SVOCs SIM including pentachlorophenol, 1,4-dioxane via SVOC SIM with isotope dilution, total and dissolved metals.
- Two (2) rounds of sampling of bedrock groundwater at newly installed bedrock well (3 ports) VOCs (full scan and SIM), SVOCs SIM including pentachlorophenol, 1,4-dioxane via SVOC SIM with isotope dilution, total and dissolved metals

The project task is described in the schedule and text below. The rationale for the specific sampling design and approach is presented in Worksheet #17.

Brush Clearing

Portions of the Site are significantly overgrown with vegetation which will have to be removed to access sample locations for the safe installation of soil borings and permanent monitoring wells.

Brush clearing will also be conducted to facilitate access for a geophysical survey consisting of seismic refraction transects along the northern fence line of the site as well as borehole utility clearance. Hand tools and mechanized equipment will be used to trim the overgrowth along the Site access road and clear paths to the sampling and geophysical locations. Brush clearing for seismic lines will be wide enough to allow personnel to walk equipment (minimum 4 feet width). Brush clearing for borehole clearance will an approximate 30 square foot area (to the extent practical) centered over the borehole. Stumps will be less than 2 to 3-inches in height to allow ground penetrating radar (GPR) equipment to have significant coupling to the ground surface. Only small shrubs, brush, and saplings will be cleared. No hardwood trees will be removed. To protect the habitat of federally threatened Northern Long-Eared Bat (*Myotis septentrionalis*), no trees or sapling with a diameter 3-inches or larger will be removed. Cut brush will be left on-Site in the general vicinity of its generation. The vegetation will be cleared prior to the initiation of field activities.

Utility Survey

Per CENAE policies, utility clearance is required for intrusive work, regardless of planned intrusive depth. Prior to intrusive activities, the FTL is responsible for marking-out planned intrusive locations and opening a ticket with the DigSafe one-call utility clearance contractor. Coordination with the Town of Foster and representatives familiar with the Site will performed to obtain information on replacement water lines and other infrastructure. Precautionary measures (e.g., geophysical survey, air knifing methods, hand-digging to 5 ft, etc.) are required if utility clearance is not confirmed. Lack of confirmation can include urban locations, areas adjacent to roadways, areas not previously assessed, areas with insufficient utility information, or areas with multiple lines. The location of utilities will be noted and recorded during the Site visits and referenced when selecting investigation locations. Utility Clearance will be conducted in accordance with AECOM *SOP 3-01: Utility Clearance* (updated SOP included in Appendix A).

Field Instrument Calibration and Quality Control

Equipment will be checked to ensure its completeness and operational readiness. Equipment found to be damaged or defective will be returned to the point of origin, and a replacement will be secured. Instruments and equipment that require route maintenance and/or calibration will be checked initially upon arrival and then prior to use each day, if needed to support the field activities scheduled for that day. Equipment calibration will be conducted in accordance with AECOM *SOP 3-20: Operational and Calibration of a PID* and AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling* (updated SOPs included in Appendix A).

This system of checks ensures that the equipment is functioning properly. If an equipment check indicates that a piece of equipment is not operating correctly and field repair cannot be made, the equipment will be tagged and removed from service, and a request for replacement equipment will be placed immediately. Replacement equipment will meet the same specifications for accuracy and precision as the equipment removed from service.

Geophysical Survey

The existing geological dataset will be supplemented for overburden thickness, weathered bedrock zone identification/depth/thickness, competent bedrock depth, and potential fracture orientation. Borehole locations will also be cleared for subsurface utilities using geophysical methods. Noted that previous subsurface utility clearance efforts conducted on the site has been deemed as suspect and shall not be relied upon for the purposes of these efforts.

Utility Clearance

For the utility clearance, three complementary geophysical methods will be utilized including time domain electromagnetic induction metal detection (EM61), ground penetrating radar (GPR), and precision utility location (PUL). The EM61 data will be acquired at approximately 8-inch intervals along lines spaced five feet apart across the accessible portions of the area of interest (AOI) where they are clear of surface metal obstructions. The EM61 survey will detect and outline areas containing buried metal. However, the EM method cannot provide information on the type of objects causing an EM anomaly. GPR data will be acquired along traverses spaced five feet apart in a grided fashion surveying a 30 square foot area centered over each boring location. The PUL method will be used to search for subsurface utilities in the accessible portions of the area of interest by passively searching for signals from active electric lines and by tracing utilities from direct connections to surface features such as valves and conduits.

For the EM61 survey, a Geonics EM61-MK2 time domain electromagnetic induction metal detector will be used. The EM61 is a time-domain electromagnetic induction type instrument designed specifically for detecting buried metal objects. An air-cored 1-meter by ½-meter transmitter coil generates a pulsed primary magnetic field in the earth, thereby inducing eddy currents in nearby metal objects. The decay of the eddy current produces a secondary magnetic field that is sensed by two receiver coils, one coincident with the transmitter and one positioned 40 cm above the main coil. By measuring the secondary magnetic field after the current in the ground has dissipated but before the current in metal objects has dissipated, the instrument responds only to the secondary magnetic field produced by metal objects. Four channels of secondary response are measured in mV and are recorded on a digital data logger. The system is generally operated by pulling the coils as a trailer with an odometer mounted on the axle to trigger the data logger

automatically at approximately 8-inch intervals. EM61 method generally penetrates up to 10 feet below ground surface.

Electromagnetic and magnetic geophysical methods, including the methods proposed here, are affected by the presence of power lines and surface metal objects (steel sided buildings, dumpsters, vehicles, reinforced concrete, etc.). Where such are present, the effects of materials in the subsurface may be masked, and firm conclusions about subsurface conditions cannot be made.

Detection and identification should be clearly differentiated. Detection is the recognition of the presence of a metal object, and the EM method is excellent for such purposes. Identification is determination of the nature of the causative body (i.e., what is the body - a drum, UST, automobile, etc.?), and EM cannot identify the buried metal object.

For the GPR survey, either a GSSI UtilityScan DF subsurface imaging radar system or GSSI SIR 4000 subsurface imaging radar system will be utilized. Data are recorded digitally, and the GPR data can be reviewed in the field. The systems include survey wheels that trigger the recording of the data at fixed intervals, thereby increasing the accuracy of the locations of features detected along the survey lines.

To clear borehole locations of utilities, the UtilityScan DF will be used to acquire data simultaneously from 800 MHz and 300 MHz antennas, or the SIR 4000 system will be used to acquire data using a 350 MHz hyperstacking antenna. To map bedrock, surface the UtilityScan DF 800 MHz and 300MHz or the 150 MHz antennas will be used. Please note that GPR penetration depends greatly on the site soil conditions.

There are limitations of the GPR technique as used to detect and/or locate targets such as those of the objectives of this survey: (1) surface conditions, (2) electrical conductivity of the ground, (3) contrast of the electrical properties of the target and the surrounding soil, and (4) spacing of the traverses. The condition of the ground surface can affect the quality of the GPR data and the depth of penetration of the GPR signal. Sites covered with high grass, bushes, landscape structures, debris, obstacles, soil mounds, etc. limit the survey access and the coupling of the GPR antenna with the ground. Boring/well locations and traverse lines will be cleared of brush and debris. Tree/bush stumps will not protrude greater than 3 inches above the surface.

The electrical conductivity of the ground determines the attenuation of the GPR signals and thereby limits the maximum depth of exploration. For example, the GPR signal does not penetrate clay-rich soils and targets buried in clay might not be detected.

A definite contrast in the electrical conductivities of the surrounding ground and the target material is required to obtain a reflection of the GPR signal. If the contrast is too small, then the reflection may be too weak to recognize, and the target can be missed. Typically, in New England the electrical conductivity contrast between soil and bedrock surface is not large enough to clearly identify bedrock surface.

The PUL survey, will use a Radiodetection RD8000 precision pipe and cable location system. The RD8000 is an electromagnetic instrument that consists of a separate transmitter and receiver. The receiver can detect subsurface utilities and cables in three modes by detecting a signal on the utility sent from the transmitter, by passively detecting signals from nearby power lines, or by passively detecting signals from distant radio transmitters.

The PUL equipment cannot detect non-metallic utilities, such as pipes constructed of unreinforced concrete, clay, ceramic, plastic, PVC when used in passive mode alone. Such pipes can be detected with the RD8000, however, where surface access permits insertion of a device on which the signal can be transmitted.

The instrument also generally cannot be used to locate metal utilities located under reinforced concrete because the signal couples onto the metal rebar and mesh in the concrete, and the signal on a particular utility cannot then be traced reliably. Similarly, the method commonly cannot be used adjacent to grounded metal structures such as chain link fences and metal guardrails.

Positively identified utilities in the vicinity of work areas will be marked out by the Subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). White or pink will be used for anomalies.

The location of utilities will be recorded by the Subcontractor using a global positioning system (GPS) unit with sub-meter accuracy combined with measurements to Site features recorded to tenths of a foot with survey tapes. Within one (1) week of completing fieldwork, the Subcontractor shall submit the raw data files and a map showing the locations of utilities, subsurface structures, and anomalies identified. The buried utility map will be submitted in draft and final versions.

Supplemental Geophysical Survey

For mapping geological subsurface layering, a seismic refraction survey along the two 300 to 500-foot transects oriented east to west will be completed along the northern fence line of the Site. A GPR survey along the transects will also be completed. (Note: historical geophysical reports and data will be share with the geophysical subcontractor prior to commencement of the work.)

For seismic refraction, a 48-channel seismograph (two 24-channel Geometrics Geodes) coupled to up to 48 14-Hz geophones will be used with geophone spacing at 5 feet for this survey. This survey will be used to evaluate weathered bedrock zone identification/depth/thickness, competent bedrock depth, and potential fracture orientation. The seismograph is connected to, and controlled by, a notebook PC computer. The software provides for the acquisition, display, plotting, filtering, and storage of seismic data. A 12-pound sledgehammer or an accelerated weight drop will be used as an energy source. Generally, five to seven "shots" per cable spread consisting of one shot off each end of the cable, one shot at each end of the cable, and one to three shots interior to the cable. This configuration provides reversed profiles. The number of stacks per shot point is variable, and the quality of the stacked seismic signal for each shot point will be verified in the field. The data will be recorded digitally.

The seismic data will be interpreted with the Generalized Reciprocal Method, commonly referred to as GRM. GRM allows the depth to bedrock to be determined for each geophone location, rather than only at the shot points as for most other methods, and it is less sensitive to the presence of dipping interfaces and hidden layers. Fracture identification/orientation can be determined if a significant drop in seismic velocity is observed within the data set.

Similar to the utility locating task, the Subcontractor will survey the locations of the transects using a Trimble GeoX7 CM PS system coupled with a Zephyr-2 external antenna. A report summarizing the geophysical methods used and results will be provided by the Subcontractor. The Subcontractor will submit annotated/interpreted 2D models of the subsurface from the geophysical data collected. A map summarizing interpreted fracture orientations, weathered zone locations, and thicknesses will be provided. Raw data and field notes will also be submitted, in addition to processed and presented data.

The seismic refraction method assumes that the local geology is uncomplicated. In particular, the seismic refraction method assumes that interfaces between geologic materials correlate with sharp increases in seismic velocity and that the interfaces between geologic units are flat lying. The method is not extremely sensitive to lateral variations within layers, and subtle features such as fracture zones within bedrock cannot be detected unless there is a topographic expression of the feature and/or a significant drop in bedrock velocity. The accuracy of the method is degraded in areas with strong topographic relief and/or where the interfaces have apparent dips greater than about 20 degrees. In general, the accuracy of depths determined is stated to be about 10% or 2 feet, whichever is greater.

Where two materials do not exhibit contrasting velocities, or where velocities gradually increase

with depth, a clear refracted signal is not generated, and the seismic refraction method cannot be used to distinguish the two materials. In some cases, the "geophysical contact" between materials with contrasting velocities does not correlate exactly with the "geologic contact." For example, where a highly weathered bedrock is overlain by a dense material such as till, the velocity range of the weathered bedrock might overlap or approach the velocity range of the till, and the two materials cannot be distinguished seismically. In such cases, the depth determined by seismic refraction is the depth of competent bedrock, which might be located at some depth below the geologic contact.

The depth relations of the water table and bedrock may constitute a significant problem for the seismic refraction technique. This problem is that of a "blind layer." A blind layer occurs where the thickness of the saturated overburden is less than about half the depth of bedrock. In such cases, the water-saturated material immediately above bedrock is "blind" in the sense that no refracted seismic energy from it will be received as a first arrival of seismic energy, and all methods used to reduce the seismic data to determine the depth of bedrock, the objective of this survey, use only first arrivals. Thus, the saturated layer will not be detected where it is close to bedrock, and most methods of seismic data reduction will indicate that bedrock is shallower than it is. Although GRM, the method used by Hager-Richter to reduce the seismic refraction data, does not use first arrivals through the water saturated zone (because there is none to use) in such cases, GRM determines the depth of bedrock correctly by using the average velocity of the saturated and unsaturated zones.

A "hidden layer" occurs where a lower velocity material underlies a higher velocity material, a common situation in stratified sediments. An example is where sands are present under layers of clay or till. As in the case of a "blind layer," most methods of seismic refraction data reduction will indicate that bedrock is deeper than it is if a hidden layer is present but not detected. Internal tests in the seismic refraction data reduction software (IXRefraX by Interpex) indicate that such layers might be present and an average velocity of the two layers is used to determine the depth of bedrock.

Soil Boring Advancement and Surface/Subsurface Soil Sampling

As part of this RI, Optional Task 14 will be executed once. The Optional Task 14 execution includes the advancement of five (5) soil borings to 20 feet below ground surface (bgs) totaling up to 100 linear feet (Note: that actual depths in the field may vary per location). The advancement of a sixth overburden soil boring using rotosonic drilling to an estimated depth of up to 35 feet below ground surface (bgs) will also be performed to facilitate the installation of a bedrock well. Up to two (2) soil samples will be collected from each of the advanced borings. The target depth is

specified for each location in Worksheet #17 of this QAPP. The recovered soil core will be screened for VOCs with a PID immediately upon opening the sleeve. The soil core will be logged for descriptions by an experienced and qualified field geologist. Observations and measurements will be recorded on a soil boring log. At a minimum, depth interval, recovery, PID concentrations, moisture, and texture using the Unified Soil Classification System (USCS) will be recorded. Additional observations to be recorded may include detectable odors, groundwater depth, organic materials, cultural debris, or color changes indicative of oxidation changes or staining. See AECOM *SOP 3-16: Soil and Rock Classification* (updated SOP included in Appendix A) and AECOM *SOP 3-21: Surface and Subsurface Soil Sampling Procedures*.

Two soil samples will be collected from each boring location unless otherwise specified in field modification documentation. One (1) soil sample will be collected from surface soil (0-2 feet) and one from the area(s) with highest PID detections, visible contamination, or most likely depth for impacts. Surface soil samples are considered to be 0 to 2 ft bgs and subsurface soil samples are considered deeper samples that indicating evidence of impacts or at the bedrock interface. VOC samples will be collected directly from the soil core using a sampling corer (e.g., Terra Core, plastic syringe with tip removed, or similar) and in accordance with AECOM SOP: 3-21. For other analyses, soil will be removed and transferred to a disposable, re-sealable plastic bag. The sample will then be mixed until the sample is a uniform color, texture, and particle size. Non-mixed particles, organic matter, or other non-soil debris will be removed. After mixing, the sample will be transferred to the appropriate sample containers for laboratory analysis. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain preservation, as appropriate. The required samples containers, preservatives, and holding times are specified in Worksheet #19 & 30. Sample locations will be marked with a pin flag labeled with the sample identification number. The locations will also be photo-documented and recorded with a hand-held GPS.

Soil sampling described above is to be considered “biased” for the purposes of filling data gaps in connection with nature and extent. Unbiased soil sampling will be included as part of a future QAPP Addendum to establish the soil dataset for risk assessment purposes.

Overburden/Weathered Bedrock Monitoring Installation and Development

Four (4) permanent overburden and/or weathered bedrock monitoring wells will be installed during the additional RI field investigation. Overburden drilling procedures are detailed above in the *Soil Boring Advancement and Surface and Subsurface Soil Sampling* section. The rationale for the selected overburden monitoring well locations is provided in Worksheet #17. The exact

depth and monitoring well construction details will depend on field observations of the geology and observation of potential impacts.

An unused 2-inch diameter milled slot PVC screen and casing will be installed to the bottom of the borehole. The sand pack material will be selected based on the grain size distribution of the surrounding aquifer and placed in lifts as the drilling crew pulls the casing back to expose the well screen. The screen slot size and length will be determined in the field based on the field observations and screening. The borehole above the sand pack will be sealed with 2 ft of hydrated bentonite chips. Water will be added if the seal is not placed within saturated material. Bentonite chips will be allowed to hydrate for one-hour prior to grouting. The remaining annular space between the well casing and the 6-inch override casing from the top of the bentonite seal to the ground surface will be pressure grouted with a bentonite/cement grout using a tremie pipe. The remaining 6-inch diameter sonic casing will be pulled from the ground. Vibration will be applied to the casing as it is pulled to densify and degas the grout, as well as knit the grout into the borehole wall, creating a superior seal.

Permanent overburden monitoring wells will be developed no sooner than 24 hours after completion of well installation. Development will be completed by a combination of surging with a surge block and over-pumping with a submersible monsoon pump or Wattera pump and associated HDPE tubing, in accordance with AECOM *SOP 3-13: Monitoring Well Development* (updated SOP included in Appendix A).

Low-flow sampling will be performed following the USEPA Guidance on low stress purging and sampling (USEPA, 2017). Water clarity will be visually monitored and water quality parameters, including dissolved oxygen (DO), specific conductance (SC), oxidation-reduction potential (ORP), pH, temperature, and turbidity will be measured using a flow-through cell every 5 minutes during purging to determine progress of development per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling*. The multi-parameter water quality meter will be calibrated at the beginning of each day. A calibration check[2] will be performed at the end of each day and anytime anomalous readings are observed. Each well will be developed until the well produces clear (silt-free) water with a minimum of 3 successive stable water quality readings as outlined below:

- pH – within ± 0.2 units.

[2] An anomalous reading is an unexpected measurement that is often accompanied with an instrument error message. For example, a creeping reading, elevated readings in ambient air, etc. that would necessitate a calibration check

- DO – within ± 10%
- SC – within ± 3 percent (%).
- ORP – within ± 10 millivolts (mV).
- Temperature – within ±1 degree Celsius.
- Turbidity – at or below 10 nephelometric turbidity unit (NTU) or within ± 10% if above 10 NTU.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry a minimum of three times in succession and the turbidity has decreased. If water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development.

Reusable sampling equipment will be properly decontaminated after each use in accordance with AECOM SOP 3-06. Excess soil or groundwater generated will be containerized, managed, and disposed of as IDW.

Bedrock Monitoring Well Installation and Development

One (1) permanent bedrock monitoring well will be installed during the additional RI field investigation. The rationale for the selected bedrock monitoring well location and target depth is provided in Worksheet #17. Drilling activities will be performed by a driller licensed by the Rhode Island and have experience using water hammer drilling methods. Health and safety consideration for this drilling technique are provided in an update to AECOM's Accident Prevention Plan. Prior to starting, the overburden and weathered bedrock units should be sealed off (this should be done by the roto-sonic drill rig team). To seal off the overburden and weathered bedrock, 5-inch permanent steel casing will be advanced 5 ft into competent rock and tremie-grouted to the surface as 8-inch temporary casing is withdrawn. The top of the 5-inch casing will be threaded to allow future attachment of a temporary casing extension at the surface, if needed during FLUTe installation. Hydraulic/water hammer drilling may proceed after a minimum 24-hour curing period.

Drilling will advance to the target depth listed in Worksheet #17. The recovered rock chips/fragments will be logged (as best as possible) for descriptions by an experienced and qualified field geologist. Where water hammer drilling techniques are being conducted, the field geologist will also observe and record water gain and/or water loss as these can be indicators of likely fracture zone(s). Water utilized for this drilling event will be provided by the drilling

contractor. Unlike the USCS for soils, there is no single standard rock classification system; however, the field geologist will describe the essential items. At a minimum, depth interval, rock classification name, color, mineralogical composition and percentage, and texture/grain size should be recorded. See AECOM *SOP 3-16: Soil and Rock Classification* and EM-1110-1-1804.

The permanent bedrock borehole will be developed at least 24 hours after completion of well installation. Development will be completed by a combination of surging with a surge block and a venturi air lift pump. Sufficient development of the newly installed bedrock well will be completed to account for the volume of water introduced to the well via water hammer drilling technique to facilitate additional follow up data collection (i.e., borehole geophysics, FLUTe testing, groundwater monitoring). The construction of the surge block will be appropriate for the 4-inch diameter borehole and be mounted on rods for downhole advancement. The borehole will be surged in 10-foot intervals followed by purging. Bedrock borehole development will be performed by the drilling subcontractor with oversight from the field geologist.

Similar to the overburden well development, water clarity will be visually monitored and water quality parameters measured using a flow-through cell every 5 minutes during purging to determine progress of development per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling*. Groundwater generated will be containerized, managed, and disposed of as IDW (see section regarding IDW management).

Bedrock Borehole Geophysics

After completion of the bedrock well development, borehole geophysical logging will be performed on the bedrock borehole. If a significant period of time is scheduled between the borehole development and geophysical logging, a blank FLUTe liner will be installed within the borehole to act as a seal and prevent intra-borehole flow. Once geophysical logging is scheduled, testing will be performed include using the following tools:

- Acoustic caliper
- Fluid temperature
- Fluid conductivity/resistivity
- Optical televiewer (OTV)
- Acoustic televiewer (ATV)
- Natural gamma ray
- Spontaneous potential/resistivity

- Heat pulse flow meter (HPFM) under ambient conditions
- HPFM under pumping conditions

The results of the geophysical logging will be one line of support evidence in the development of the final FLUTe Multi-Level Sampler (MLS) liners.

FLUTe Installation

Following completion of the geophysical logging, a blank FLUTe liner will be installed in the bedrock borehole. The blank FLUTe liner will be equipped with FLUTe Activated Carbon Technique (FACT) covered with felt strips. The FACT strips will remain in the borehole for a minimum period of two weeks. After this wait period, the FACT strips will be removed and visually inspected and screened with a PID. The FACT strip will be cut into three-foot segments, bottled, and shipped to a laboratory subcontracted by FLUTe for analysis. The FACT strip will be labeled with the borehole identification and placed in a clean trash bag for on-Site storage.

A piece of the FACT liner will be used as a field blank. This sample will be collected from a FACT liner that does not contact groundwater to provide a check on liner handling and potential cross-contamination. Two such blanks will be collected - one prior to liner deployment and another at the time of sample retrieval, collected from a depth above the water table.

As a final test, the FLUTe blank liner will be reused to conduct a transmissivity and reverse head profile in the bedrock borehole. Upon completion of the transmissivity and reverse profiling, the blank FLUTe liner will remain in the borehole to act as a seal and prevent intra-borehole flow until the blank FLUTe liner can be replaced. Borehole information obtained during the FLUTe testing will be compiled and processed by FLUTe. The specific procedure for the FLUTe transmissivity testing was provided in the FLUTe SOPs included in the 2020 QAPP.

Results of the borehole geophysical logging will be combined with FLUTe transmissivity and reverse head profiles, and FLUTe FACT strip analytical results. The objectives of the geophysical logging will be to identify potential water-bearing fracture zones in the bedrock along the length of each open hole, to define fracture depths, strikes, and dips, and to assess the actual and potential inflow/outflow of a subset of those fractures. A secondary goal is identifying changes in bedrock lithology. The FLUTe testing will provide information regarding the transmissivity of the borehole every foot and the relative distribution of dissolved phase VOCs in the borehole.

The team will review the integrated geophysical and FLUTe borehole data and determine the FLUTe MLS liner construction details for the bedrock monitoring well. FLUTe will then construct the MLS liner in accordance with the specifications provided. The FLUTe MLS liner will include

three (3) sampling ports. If warranted and based on the review of the integrated well logs, additional ports may be included in the design and construction of the MLS liner. FLUTe work will be performed by qualified personnel trained by FLUTe in the installation and operation of their equipment.

Synoptic Groundwater Gauging

Groundwater levels will be used to monitor Site-wide groundwater elevations and assess groundwater flow in the overburden and bedrock groundwater zones. Synoptic groundwater gauging rounds are anticipated to take place in spring and fall at site wells prior to purging and sampling. Synoptic water level elevation measurements will be collected from the overburden monitoring wells, piezometers, and bedrock FLUTe ports from the survey measurement point using a water level meter (Solonist 101 or equivalent). Synoptic gauging will include stream gauging station PR79-STATION-001. Previous stream gauge location PR79-STATION-002 was lost to potential storm damage. An alternate stream gauging location has been identified as the United States Geological Survey (USGS) stream gauge on Windsor Brook located at Windsor Road (USGS monitoring location 01115185). Steam gauge location 01115185 was established in 1993 located at latitude/longitude 41.83621005, -71.72256829 (NAD83) within the Narragansett subbasin. The measured elevation is 397.5 ft amsl (NAVD88).

New Overburden Groundwater Sampling

Overburden and weathered bedrock groundwater samples will be collected from the four (4) newly installed monitoring wells. The complete list of monitoring wells is provided in Worksheet #17 and shown on Figures 17-1A and 1B.

Prior to sampling, groundwater levels will be measured in each well using a water level meter (Solonist 101 or equivalent). The monitoring wells will be purged following low-flow sampling techniques using a bladder or peristaltic pump and disposable tubing in accordance with AECOM *SOP 3-14: Monitoring Well Sampling* (updated SOP included in Appendix A). Water clarity will be visually monitored and water quality parameters, including dissolved oxygen (DO), specific conductivity (SC), oxidation-reduction potential (ORP), pH, and temperature will be measured using a flow-through cell per the AECOM *SOP 3-24: Water Quality Parameter Testing for Groundwater Sampling*. Turbidity measurements will be collected using a separate turbidity meter. Turbidity samples are collected before the flow-through-cell. A “T” connector (or equivalent) coupled with a valve is connected between the pump’s tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed, and the container sample is then placed in the turbidity meter. Readings will be collected every 5 minutes until the well produces clear (silt-free) water

for a minimum of 3 stable water quality readings, as outlined above in SOP 3-14. The stabilization requirements are provided in SOP 3-14. The multi-parameter water quality meter and turbidity meter will be calibrated at the beginning of each day. A calibration check will be performed at the end of each day and anytime anomalous readings are encountered. Non-disposable sampling equipment will be decontaminated between each well per AECOM *SOP 3-14: Monitoring Well Sampling*.

Once the water quality parameters reach stabilization, field samples will be collected into laboratory-supplied bottleware for the methods listed in Worksheets #18 and #20 as (refer to Worksheet #19 & 30 for preservation, holding time, and bottleware requirements). Samples requiring filtering will be field filtered. In addition to the normal samples, quality control samples consisting of field duplicates, matrix spike (MS), and matrix spike duplicate (MSD) will be collected as outlined in Worksheet #20. One trip blank (TB) will be submitted each day if normal VOC samples are collected. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain a preservation, as appropriate. Samples will be quality-control checked by the field team (label correctness, completeness, etc.) and recorded on Chain-of-Custody (CoC) forms. Samples will be packaged on ice and transported via overnight commercial carrier or a laboratory courier under standard chain-of-custody procedures to the laboratory.

New Bedrock Groundwater Sampling

Bedrock groundwater samples will be collected from the newly installed FLUTe bedrock well (3 sampling ports). The monitoring well and sampling ports are provided in Worksheet #17 and shown on Figure 17-1B.

Groundwater sampling procedures have been developed by FLUTe based on the transmissivity testing performed during the FLUTe installation. Prior to sampling, the water level inside the FLUTe liner will be gauged to ensure water has not been lost since the previous sampling round. Water will be added, if needed, to raise the water level to the specified head (as per FLUTe guidance). Next, the water level within each sampling port will be gauged using a Solinst 102 P10 (or similar). After completion, the nitrogen gas will be connected via a regulator to the FLUTe sampling manifold. The specific sampling gas pressure (determined by FLUTe) will be used during the purge and sampling cycles. Per FLUTe guidance, the pump and sample tube should be purged at the specific purge pressure four times prior to sampling. After this, sampling at the designated sample pressure can begin on the fifth cycle. The specific sampling procedures for the FLUTe bedrock well is included in the FLUTe SOP in Appendix D of the original QAPP March 2020). Samples will be collected into laboratory-supplied bottleware for the methods listed in

Worksheets #18 and #20 as (refer to Worksheet #19 & 30 for preservation, holding time, and bottleware requirements). Samples requiring filtering will be field filtered. In addition to the normal samples, quality control samples consisting of field duplicates, MS, and MSD will be collected as outlined in Worksheet #20. One TB will be submitted each day if normal VOC samples are collected. Sample jars will be labeled with the appropriate information, placed in a Ziploc bag, and stored in a cooler containing bagged ice to maintain preservation, as appropriate. Samples will be quality-control checked by the field team (label correctness, completeness, etc.) and recorded on CoC forms. Samples will be packaged on ice and transported via overnight commercial carrier or a laboratory courier under standard chain-of-custody procedures to the laboratory.

Investigation Derived Waste (IDW) Management

IDW generated during field activities will be managed pursuant to applicable Federal, State, and local regulations and guidance, including USACE guidance (2013) and RIDEM Policy Memo 95-01 Guidelines for the Management of Investigative Derived Waste (RIDEM, 1995). Refer to AECOM *SOP 3-05: Investigation-Derived Waste Management* for procedures related to IDW management. Department of Transportation (DOT) compliant shipping containers will be used to stage IDW prior to off-Site transport. Solid IDW (e.g., drill cuttings from boring/monitoring well installation that cannot be returned to the borehole of origin) will be stored in 55-gallon metal drums and/or a 20 cubic yard closed-top roll-off bin; liquid IDW (e.g., monitoring well development water, purge water, decontamination water, drilling water) will be stored in frac tanks (or equivalent) and/or 55-gallon metal drums.

The IDW containers will be properly labeled, sampled for waste characterization, and temporarily staged on Site at a designated secure location until waste characterization is completed in accordance with applicable Federal and State guidance and regulations. The IDW containers will subsequently be transported to the approved off-Site disposal facility; the intended facility will confirm their acceptance of the waste prior to transport. IDW removal from the Property will be documented by manifest or bill of lading prepared by the waste disposal subcontractor.

QAPP Worksheet #17: Sampling Design and Rationale

This worksheet describes the sampling design and basis for selection for the soil, overburden groundwater, and bedrock groundwater sample locations.

17.1 Soil Sampling

Discrete surface and subsurface soil samples will be collected in areas with identified data gaps within the established AOCs. The analytical methods for soils are shown in Worksheet #18 and

include VOCs (full scan), SVOCs SIM, and total metals. The locations of the proposed soil borings were determined after evaluating sampling results from previous investigations. A total of six (6) soil borings will be advanced as indicated below. The soil boring locations are shown on Figures 17-1A and 17-1B. Two soil samples will be collected from each soil boring: one surface soil sample (collected from the 0-2 ft bgs interval) and one subsurface soil sample (collected no deeper than 10 ft bgs). The sample intervals were specifically selected for several reasons:

- Surface soils collected during the Phase I Site investigation were collected from the 0-2 ft bgs interval. For consistency and to combine the results of both datasets, the RI surface soil samples will be collected from 0-2 ft bgs (see Table 17-1 for rationale).
- Due to the Site history (construction, activities, and demolition), it was agreed that sampling 0-1 ft bgs interval would likely only target reworked soils and not reach native material.
- Surface soils will be collected from the 0-2 ft bgs interval to allow for comparisons to historic datasets.
- Subsurface soil samples will not be collected from depths greater than 10 ft bgs, which is the maximum depth to which human receptors may be exposed. Field screening will be performed to determine the exact sampling depth (highest PID, water table elevation, or lithologic interface) within the 2-10 ft bgs interval.
- Soil borings will be field screened for the entire length of the boring (to a maximum depth of boring). Additional subsurface samples will be collected if an interval of interest is observed (non-aqueous phase liquid [NAPL] present, elevated PID reading, or lithologic interface).

The proposed soil boring samples are listed in Table 17-1 along with the rationale for the selected location. Soil sampling procedures are summarized in Worksheet #14. Specific sample analyses are provided in Worksheet #18.

Table 17-1: Sampling Design and Rationale for Soil

AOC	Location ID	Depth	Rationale
AOC-1	PR79-SB-141 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located along the northern property boundary. Additional sampling is required for horizontal and vertical data coverage along the northern property boundary.
AOC-1	PR79-SB-142 (Figure 17-1A)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located along the northern property boundary. Additional sampling is required for horizontal and vertical data coverage along the northern property boundary.
AOC-3	PR79-SB-143 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located between the former heating oil UST and former septic tank. Limited soil data exists in this area from previous investigations. Additional sampling is required for horizontal and vertical data coverage.
AOC-3	PR79-SB-144 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Replacement soil boring, formerly SB112, that will be moved away from the existing septic tank to a location between the septic tank and the property boundary at AOC-3.
AOC-3	PR79-SB-145 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located north of the seep near porewater sample location WT-007 along the Pipeline and Service Pathway from the Distribution Box to the Sand Pits and Chlorine Chamber. Intercept midpoint for COPCs migrating from the hilltop to Winsor Brook. Additionally, sewage leaking from the pipeline could potentially impact underlying soil and the pipeline bed could act as a preferential pathway for COPCs to flow. Further investigation is warranted to investigate this identified data gap.
23B Theodore Foster Road	PR79-SB-146 (Figure 17-1B)	Surface: 0-2 ft bgs Subsurface: up to 10 ft bgs	Located downgradient of drinking water wells ROU-2 and ROU-3. Soil boring will be advanced to facilitate the installation of an interceptor bedrock well.

17.2 Overburden and Bedrock Groundwater Sampling

The primary goal for the monitoring well sampling is to select locations that targeted data gaps in the overburden, weathered bedrock, and bedrock where potential constituents of potential concern (COPCs) could be acting as a continuing source to the dissolved concentrations observed in downgradient water supply wells. Two rounds of sampling of the newly installed monitoring wells is being conducted for VOCs (full scan and SIM), SVOCs including pentachlorophenol (SIM), 1,4-dioxane[3] (SIM via isotope dilution), and total metals, and dissolved metals (field filtered) analysis to bolster the data set for the risk assessment. Table 17-2 provides the rational for the locations included in the groundwater sampling program. Sample locations are shown on Figures 17-1A and 1B.

[3] Based on the Phase I Risk Screening results, 1,4-Dioxane was detected in groundwater wells sampled during the December 2020 sampling event at concentrations greater than the USEPA RSL for tapwater, including BR-001, BR-002, BR-003, BR-004, and BR-005.

Table 17-2: Sampling Design and Rationale for Overburden and Bedrock Groundwater

AOC	Location ID	Screen Interval	Rationale
Overburden and Weathered Bedrock Monitoring Wells			
AOC-1	PR79-MW-009	TBD	Assess groundwater flow north of AOC-1 (Former Radar Pad B) and evaluate if TCE is migrating to the north in overburden groundwater.
AOC-1	PR79-MW-010	TBD	Assess groundwater flow north of AOC-1 (Former Radar Pad B) and evaluate if TCE is migrating to the north in overburden groundwater.
AOC-3	PR79-MW-011	TBD	Assess the potential source of TCE detected at the seep at sampling location WT007, located southwest of AOC-3.
AOC-3	PR79-MW-012	TBD	Assess overburden groundwater between the former septic tank and the southwestern property boundary.
Bedrock Monitoring Wells			
AOC-1	PR79-BR-006	3 sample ports (depths TBD)	Assess bedrock groundwater downgradient from drinking water wells ROU-2 and ROU-3, south of the Site.

QAPP Worksheet #18: Sampling Locations and Methods

Soil

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
Soil							
PR79-SB-141	PR79-SB-141-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-142	PR79-SB-142-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-143	PR79-SB-143-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-144	PR79-SB-144-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-145	PR79-SB-145-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-146	PR79-SB-146-XX	Soil	0-2, up to 10	Normal	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	
PR79-SB-143	PR79-SB-FD-03	Soil	0-2, up to 10	Field QC	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	Field Duplicate
PR79-SB-142	PR79-SB-FD-04	Soil	0-2, up to 10	Field QC	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	Field Duplicate
PR79-SB-144	TBD	Soil	0-2, up to 10	Field QC	VOCs (full scan, 8260C), SVOCs (SIM, 8270D), and total metals (6010C/7471B)	3-21	MS/MSD

Overburden and Bedrock Groundwater

Sample Location (LOC ID)	Field Sample ID[1]	Matrix[2]	Depth Interval (ft bgs)	Type	Analyte / Analytical Group[3]	Sampling SOP	Comments
PR79-MW-009	PR79-MW-009-01	GW	TBD	Normal	VOCs (full scan and SIM, 8260C), SVOCs including pentachlorophenol (SIM, 8270D), 1,4-dioxane (SIM via isotope dilution, 8270E), metals (filtered and non-filtered) (6010C/6020A/7470A)	3-14	Overburden
PR79-MW-010	PR79-MW-010-01	GW	TBD	Normal		3-14	Overburden
PR79-MW-011	PR79-MW-011-01	GW	TBD	Normal		3-14	Overburden
PR79-MW-012	PR79-MW-012-01	GW	TBD	Normal		3-14	Overburden
PR79-MW-013	PR79-MW-013-01	GW	TBD	Normal		3-14	Overburden
PR79-BR-006	PR79-BR-006-01	GW	TBD	Normal	VOCs (full scan and SIM, 8260C), SVOCs including pentachlorophenol (SIM, 8270D), 1,4-dioxane (SIM via isotope dilution, 8270E), metals (filtered and non-filtered) (6010C/6020A/7470A)	3-14	Bedrock
	PR79-BR-006-01	GW	TBD	Normal		3-14	Bedrock
	PR79-BR-006-01	GW	TBD	Normal		3-14	Bedrock
PR79-MW-03	PR79-GW-FD09	GW	--	Field QC	VOCs (full scan and SIM, 8260C), SVOCs including pentachlorophenol (SIM, 8270D), 1,4-dioxane (SIM via isotope dilution, 8270E), metals (filtered and non-filtered) (6010C/6020A/7470A)	3-14	Field Duplicate
PR79-BR-006	PR79-GW-FD010	GW	--	Field QC		3-14	Field Duplicate
PR79-MW-009	TBD	GW	TBD	Field QC		3-14	MS/MSD
PR79-MW-010	TBD	GW	TBD	Field QC		3-14	MS/MSD
Field QC	GW-EB-03	AQ	NA	Field QC		--	Equipment Blank
Field QC	TB-01-03	AQ	NA	Field QC	VOCs (full scan and SIM, 8260C)	--	-02, -03, etc. if multiple collected in one day

[1]Sample IDs for samples that are field-filtered will be appended with "-F".

[2]Key: GW = groundwater, AQ=Aqueous

[3]See Worksheet #20 for full list of VOC, SVOC, and metals sampling suites.

MS/MSD = matrix spike/matrix spike duplicate
QC = quality control
SIM = selective ion monitoring
SVOCs = semi-volatile organic compounds
VOCs = volatile organic compounds
TBD = To Be Determined based on field observations
XX = Sample Interval beginning with -01, -02, -03, etc.

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

Laboratory (Name, sample receipt address, POC, e-mail, and phone numbers)[6]: Refer to table below

List any required accreditations/certifications: DoD ELAP or Rhode Island State Certification[7]

Back-up Laboratory: None

Sample Delivery Method: Overnight Courier

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Accreditation / Expiry Date	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
Soil								
Volatile Organic Compounds	SW-846 5035, 8260C/ CA-202, CA-220	ELAP, 02/01/2025	3 x 40-mL VOA vials	≤6°C 2 vials with 5 mL of volatile organic analysis (VOA)-free water, To Lab: Cool 4 ± 2°C, At lab: Freeze -20 ± 10°C 1 vial with 10 mL of Methanol Error range = ± 10% range for soil mass	NA	14 days	21 days	Katahdin Analytical Services, LLC
Semi-volatile Organic Compounds	SW846 3510C, 3520C, 8270D SIM/ CA-213	ELAP, 02/01/2025	4 oz amber jar	≤6°C,	14 days	30 days	21 days	Katahdin Analytical Services, LLC

Matrix/Analyte/Analyte Group[1]	Method/SOP[2]	Accreditation / Expiry Date	Container(s) (number, size & type per sample)[3]	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround[4]	Laboratory[5]
6010C/6020A Metals	SW846 3010A, 6010C/6020A, CA-605/CA-608	ELAP, 02/01/2025	4 oz glass jar	≤6°C	NA	6 months	21 days	Katahdin Analytical Services, LLC
Mercury	SW7471B/ CA-611	ELAP, 02/01/2025	4 oz glass jar	≤6°C	NA	28 days	21 days	Katahdin Analytical Services, LLC
Groundwater								
Volatile Organic Compounds (full scan and SIM)	SW-846 5035, 8260C/ CA-202, CA-220	ELAP, 02/01/2025	3 x 40-mL VOA vials	≤6°C but not frozen HCl to pH < 2	NA	14 days	21 days	Katahdin Analytical Services, LLC
Semi-volatile Organic Compounds (SIM)	SW846 3510C, 3520C, 8270D SIM/ CA-213, CA-502	ELAP, 02/01/2025	2 x 1-L amber glass bottles	≤6°C but not Frozen	7 days	40 days from extraction	21 days	Katahdin Analytical Services, LLC
6010 Metals (total or field-filtered)	SW846 3010A, 6010C/ CA-604, CA-608	ELAP, 02/01/2025	1 x 250-mL polyethylene bottle	HNO3 to pH < 2	NA	6 months	21 days	Katahdin Analytical Services, LLC
6020 Metals (total or filed-filtered)	SW846 3010A, 6020A/ CA-604, CA-627	ELAP, 02/01/2025	1 x 250-mL polyethylene bottle	HNO3 to pH < 2	NA	6 months	21 days	Katahdin Analytical Services, LLC
Mercury (total or field-filtered)	SW7470A/ CA-615	ELAP, 02/01/2025	1 x 250-mL polyethylene bottle	HNO3 to pH <2	NA	28 days	21 days	Katahdin Analytical Services, LLC
1,4-dioxane	USEPA 8270E-SIM / SOP 2164	ELAP, 05/30/2023	2 x 250-mL bottles	≤6°C but not frozen	7 days	40 days	21 days	Alpha Analytical, Inc.

Notes:
1 - Refer to Worksheet #15 for specific target analytes.
2 - Refer to the Analytical SOP References table (Worksheet #23).

3 - Sample containers and mass/volume required for analyses to be conducted by one laboratory may be consolidated.
4 - Turn Around Time (TAT) is presented in calendar days unless otherwise indicated.
5 - No backup laboratories have been identified. All laboratories are subcontracted by AECOM unless otherwise indicated.
6 - Katahdin Analytical Services, LLC, 600 Technology Way, Scarborough, Maine 04074. Point of Contact: Heather Manz, hmanz@katahdinlab.com, Direct – (207) 874-2400 x17, Fax – (207)775-4029. Katahdin will perform analysis of any IDW generate during sampling.
7 - Alpha Analytical, Inc., 320 Forbes Boulevard, Mansfield, MA 02048, Point of Contact, Liz Porta, eporta@alphalab.com, Direct – (508) 844-4124, Main – (508) 989-9220
8 - DoD ELAP is held for all analyses and analytes for Katahdin maintains appropriate state certification.
9 - Sodium sulfite is required only for chlorinated samples. According to the laboratory, if chlorination status cannot be confirmed prior to sampling, this dechlorinating agent should be added.

QAPP Worksheet #20: Field Quality Control Summary

		FIELD SAMPLES				LABORATORY SAMPLES		
Matrix/Parameter	Analytical Method	Field Samples	Equipment Blank	Trip Blanks[1]	Field Duplicates[2]	MS[3]	MSD/ MD[3]	TOTAL ANALYSES[4]
Soil								
VOCs	SW846 8260C	12	1	1	2	1	1	16
SVOCs SIM	SW846 8270D SIM	12	0	0	2	1	1	16
Total metals	SW846 6010C/6020A/7470A	12	0	0	2	1	1	16
Overburden Groundwater								
VOCs	SW846 8260C	8	2	2	1	1	1	16
VOCs SIM	SW846 8260C SIM	8	2	2	1	1	1	16
SVOCs SIM	SW846 8270D SIM	8	2	0	1	1	1	14
1,4-dioxane (Isotope Dilution)	SW846 8270E SIM	8	2	0	1	1	1	14
Total metals	SW846 6010C/6020A/7470A	8	2	0	1	1	1	14
Filtered metals (field filtered)	SW846 6010C/6020A/7470A	8	2	0	1	1	1	14
Bedrock Groundwater								
VOCs	SW846 8260C	6	2	2	1	1	1	14
VOCs SIM	SW846 8260C SIM	6	2	2	1	1	1	14
1,4-dioxane (Isotope Dilution)	SW846 8270E SIM	6	2	0	1	1	1	12
SVOCs SIM	SW846 8270D SIM	6	2	0	1	1	1	12
Total metals	SW846 6010C/6020A/7470A	6	2	0	1	1	1	12
Filtered metals (field filtered)	SW846 6010C/6020A/7470A	6	2	0	1	1	1	12

Matrix/Parameter	Analytical Method	FIELD SAMPLES				LABORATORY SAMPLES		TOTAL ANALYSES[4]
		Field Samples	Equipment Blank	Trip Blanks[1]	Field Duplicates[2]	MS[3]	MSD/ MD[3]	
IDW (soil)								
TCLP VOCs	SW846 8260C	1	0	0	0	0	0	1
TCLP SVOCs	SW846 8270D	1	0	0	0	0	0	1
RCRA Metals	SW846 6010C	1	0	0	0	0	0	1
PCBs	SW846 8082A	1	0	0	0	0	0	1
Flashpoint	SW846 1010A	1	0	0	0	0	0	1
pH	SW846 9045C	1	0	0	0	0	0	1
Reactivity	SW846 Ch. 7.3.1.2	1	0	0	0	0	0	1
TSS/TDS	SM2540 C &D	1	0	0	0	0	0	1
IDW (aqueous)								
VOCs	SW846 8260C	1	0	0	0	0	0	1
SVOCs	SW846 8270D	1	0	0	0	0	0	1
RCRA Metals	SW846 6010C	1	0	0	0	0	0	1
PCBs	SW846 8082A	1	0	0	0	0	0	1

Notes:
1 - One per cooler containing VOC samples. Assumes 1 cooler of VOC samples shipped per day of sampling.
2 - Ten percent of field samples, not including field QC samples, for the analyses and matrices shown.
3 - Five percent of field samples (not including field QC samples) per medium for methods required.
4 - Total includes MS, MSD, or MD.
MD - matrix duplicate
MS - matrix spike
MSD - matrix spike duplicate
PCB - polychlorinated biphenyl
SVOCs - semivolatile organic compounds
TCLP - toxicity characteristic leaching procedure
TSS/TDS - total suspended solids/total dissolved solids
VOCs - volatile organic compounds

QAPP Worksheet #23: Analytical Standard Operating Procedures Table

Laboratory/ SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM[1]	Modified for Project Work? (Y/N)
Katahdin Analytical Services						
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 06/21, Revision 22.	Definitive	Water and Soil/ VOCs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	No Variance	N
CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified for Selected Ion Monitoring (SIM), 07/21, Revision 16.	Definitive	Water and Soil/ SVOCs and PAHs	GC/MS	No Variance	N
CA-214	Closed-System Purge-And-Trap and Extraction for Volatile Organics In Soil And Waste Samples Using SW846 Method 5035, 01/20, Revision 8.	Definitive	Soil/ VOCs	Not applicable (extraction)	No Variance	N
CA-220	Analysis of Volatile Organic Compounds by Purge and Trap GC/MS SW-846 Method 8260 – Modified For Selected Ion Monitoring (SIM), 06/20, Revision 16.	Definitive	Aqueous / VOCs	GC/MS	No Variance	N
CA-226	Analysis of SVOAs by Capillary Column GC/MS: SW-846 Method 8270D, 06/17, Revision 10. (Reviewed 01/19)	Definitive	Water and Soil/ SVOCs	GC/MS	No Variance	N
CA-502	Preparation of Aqueous Samples for Extractable Semivolatile Analysis, 03/19, Revision 12.	Definitive	Water / SVOCs and PAHs	Not applicable (extraction)	No Variance	N
CA-512	Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis, 04/19, Revision 14.	Definitive	Soil/ SVOCs and PAHs	Not applicable (extraction)	No Variance	N
CA-526	Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semi-volatile Analysis, 07/20, Revision 13.	Definitive	Soil/ SVOCs and PAHs	Not applicable (extraction)	No Variance	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals, 03/22, Revision 11.	Definitive	Water / TAL Metals and TAL Metals + Boron	Not applicable (digestion)	No Variance	N

Laboratory/ SOP Number	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM[1]	Modified for Project Work? (Y/N)
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 01/19, Revision 8.	Definitive	Soil/ TAL Metals	Not applicable (digestion)	No Variance	N
CA-608	Trace Metals Analysis By ICP-AES Using EPA Method 6010, 09/22, Revision 22.	Definitive	Water and Soil/ TAL Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	No Variance	N
CA-611	Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471, 01/19, Revision 12.	Definitive	Soil/ Mercury	Mercury Analyzer	No Variance	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470, 09/22, Revision 17.	Definitive	Water / Mercury	Mercury Analyzer	No Variance	N
CA-620	Synthetic Precipitation Leaching Procedure (SPLP) for Inorganic and Non-Volatile Organic Analytes, 01/20, Revision 7	Definitive	Soil / Metals	Not Applicable (leaching)	No Variance	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 02/21, Revision 15.	Definitive	Water and Soil / TAL Metals and TAL Metal	ICP-MS	No Variance	N
SD-902	Sample Receipt and Internal Control, 01/19, Revision 13.	NA	NA	NA	NA	N
SD-903	Sample Disposal, 09/17, Revision 6. (Reviewed 01/19)	NA	NA	NA	NA	N

Notes:

1. Laboratory address, point of contact and contact information is presented in the footnotes of Worksheet #19 & 30.
2. The laboratory holds current DoD ELAP accreditation for all definitive analyses presented that are included in the DoD QSM. Certificates are presented in Appendix B.

GC/MS = gas chromatography;/mass spectrometry

QAPP Worksheet #24: Analytical Instrument Calibration Table - Katahdin Analytical Services, LLC

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS-VOCs	Initial Calibration (ICAL) - A minimum 5-point initial calibration is required for all VOCs.	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Analyst, Department Manager	CA-202
	Establish Retention Time Window Position	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	Second Source ICAL Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	4-Bromofluorobenze (BFB) Tune	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or decafluorotriphenylphosphine (DFTPP) from method.	Retune instrument and verify.	Analyst, Department Manager	
GC/MS (full scan) SVOCs GC/MS (SIM) PAHs, Pentachlorophenol and 1,4-Dioxane	ICAL - A minimum 5-point calibration is required for all SVOCs.	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Analyst, Department Manager	CA-213, CA-226
	Establish Retention Time Window Position	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Department Manager	
	Evaluation of RRT	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	CCV	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	DFTPP Tune	Every 12 hours	Criteria listed in Section 7.4, current revision of SOPs CA-213 and CA-226	Retune and/or clean source.	Analyst, Department Manager	
ICP-AES – Metals	ICAL - 1 point calibration plus blank	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-608
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110% for all project constituents.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Calibration Blank (CB)	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze CB. All samples following the last acceptable CB must be reanalyzed.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project constituents.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	Low-level Calibration Check Standard (if using one-point ICAL)	Daily after one-point ICAL.	%R must within 80%-120% for all project constituents.	Correct problem, then reanalyze.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	
ICP-MS – Metals	Tune	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value. Resolution must be <0.9 amu full width at 10% peak height. Four injections %RSD must be <5%.	Perform necessary equipment maintenance.	Analyst, Department Manager	CA-627

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	ICAL - 1 point calibration plus blank	Daily ICAL prior to sample analysis.	One point calibration plus a blank per manufacturer's guidelines.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	
	ICV	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% for all project constituents.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	After every 10 samples and at the end of each run sequence.	%R must be within 90-110% for all project constituents.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	ICS - ICSA & ICSB	Daily, before sample injections	ICSA recoveries must be less than the absolute value of the LOD and ICS-AB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	
Mercury analyzer	ICAL - 5 points plus a CB	Upon instrument receipt, major instrument change, at the start of each day.	Correlation coefficient (r) must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-611, CA-615
	ICV	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110%	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	CCB	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze CB. All samples following the last acceptable CB must be reanalyzed.	Analyst, Department Manager	
	CCV	Beginning and end of each run sequence and every 10 samples.	%R must be within 90-110%	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	CCV	At beginning of run, after every 10 samples and at the end of the run	80-120 %R	Reanalyze all samples back to last acceptable CCV recovery	Analyst, Department Manager	

Notes:

1. Refer to the Analytical SOP References table (Worksheet 23#).
2. LOD/LOQ verification procedures presented in the DoD QSM will be modified for this project in the following manner: Katahdin will make an effort to prioritize analysis of the LOD and LOQ verifications for the analyses in this project such that LODs and LOQs are verified prior to sample analysis. If LOD/LOQ verification cannot be analyzed for the quarter prior to sample receipt in that quarter, the required LOD/LOQ verification will be analyzed with the project samples. If LOD/LOQ verification is not successful but batch QC requirements are otherwise met, the data will be reported with and the failed LOD/LOQ verification will be identified in the case narrative. AECOM will then consider the verification failure as part of data validation (Refer to WS #36).

ICS = Interference Check Solution
LOD – Limit of Detection

QAPP Worksheet #28-1: Analytical Quality Control and Corrective Action –Volatile Organic Compounds, Full Scan

Matrix	Aqueous and Solids
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260C/ CA-202

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoro-methane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluoro-benzene	QSM Appendix C limits	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	QSM Appendix C Limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids					
Analytical Group	VOCs					
Analytical Method/ SOP Reference	SW-846 8260C/ CA-202					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ. A list of acceptance ranges for LOQ verification is provided in Appendix C.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank
QSM – Quality Systems Manual

QAPP Worksheet #28-2: Analytical Quality Control and Corrective Action – Volatile Organic Compounds, SIM

Matrix	Aqueous
Analytical Group	VOCs SIM
Analytical Method/ SOP Reference	SW-846 8260CSIM/ CA-220

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene (BFB)	Laboratory Limits: 70-130%R	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	Laboratory Limits: 70-130%R	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	Same as LCS RPD of all analytes ≤20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous					
Analytical Group	VOCs SIM					
Analytical Method/ SOP Reference	SW-846 8260CSIM/ CA-220					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ. A list of acceptance ranges for LOQ verification is provided in Appendix C.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank

QAPP Worksheet #28-3: Analytical Quality Control and Corrective Action – Semivolatile Organic Compounds, Full Scan and SIM

Matrix	Aqueous and Solids
Analytical Group	SVOCs and SVOCs SIM
Analytical Method/ SOP Reference	SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Full Scan - 6 per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14 SIM 2-Methylnaphthalene-d10 Fluorene-d10 Pyrene-d10 2,4-dibromophenol-d3	QSM Appendix C limits and in-house limits for 2-Methylnaphthalene, Fluorene-d10, Pyrene-d10, and 2,4-Dibromophenol-d3: 10-130 (Aq); 20-116 (Soil)	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	QSM Appendix C Limits and in-house limits for 1,4-Dioxane (30-150% Soil); (10-93% Aq), Benzoic acid (10-70% Soil); (10-151% Aq), Phenol (10-78% Aq)	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids					
Analytical Group	SVOCs and SVOCs SIM					
Analytical Method/ SOP Reference	SW-846 8270D (full scan), 8270D SIM/ CA-213, CA-226					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD	One per sample delivery group (SDG) or every 20 samples.	QSM Appendix C Limits , and in-house LCS limits For 1,4-Dioxane, Benzoic acid, and Phenol (Aq) RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Six per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results between DL and LOQ. A list of acceptance ranges for LOQ verification is provided in Appendix C.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank
QSM – Quality Systems Manual

QAPP Worksheet #28-5: Analytical Quality Control and Corrective Action – Metals and SPLP Metals (ICP-AES)

Matrix	Aqueous and Solids
Analytical Group	Metals, SPLP Metals (ICP-AES)[1]
Analytical Method/ SOP Reference	SW-846 6010C / CA-608

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	QSM Appendix C Limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous and Solids					
Analytical Group	Metals, SPLP Metals (ICP-AES)[1]					
Analytical Method/ SOP Reference	SW-846 6010C / CA-608					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ. Acceptance range for all metals LOQ verification is 67-133%R.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

1. Hardness is calculated from results generated by this analysis. No additional QC acceptance limits or MPCs apply to hardness.

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QAPP Worksheet #28-6: Analytical Quality Control and Corrective Action – Metals (ICP-MS)

Matrix	Aqueous					
Analytical Group	Metals (ICP-MS)					
Analytical Method/ SOP Reference	SW846 6020A / CA-627					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	QSM Appendix C Limits	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	Aqueous					
Analytical Group	Metals (ICP-MS)					
Analytical Method/ SOP Reference	SW846 6020A / CA-627					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD	One per preparatory batch.	QSM Appendix C Limits RPD of all analytes ≤ 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution (not applicable for rinsate blanks)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30-120% of that of initial calibration standard.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Per the method, for each sample, IS intensity must be ≥ 70% of that of initial calibration standard.

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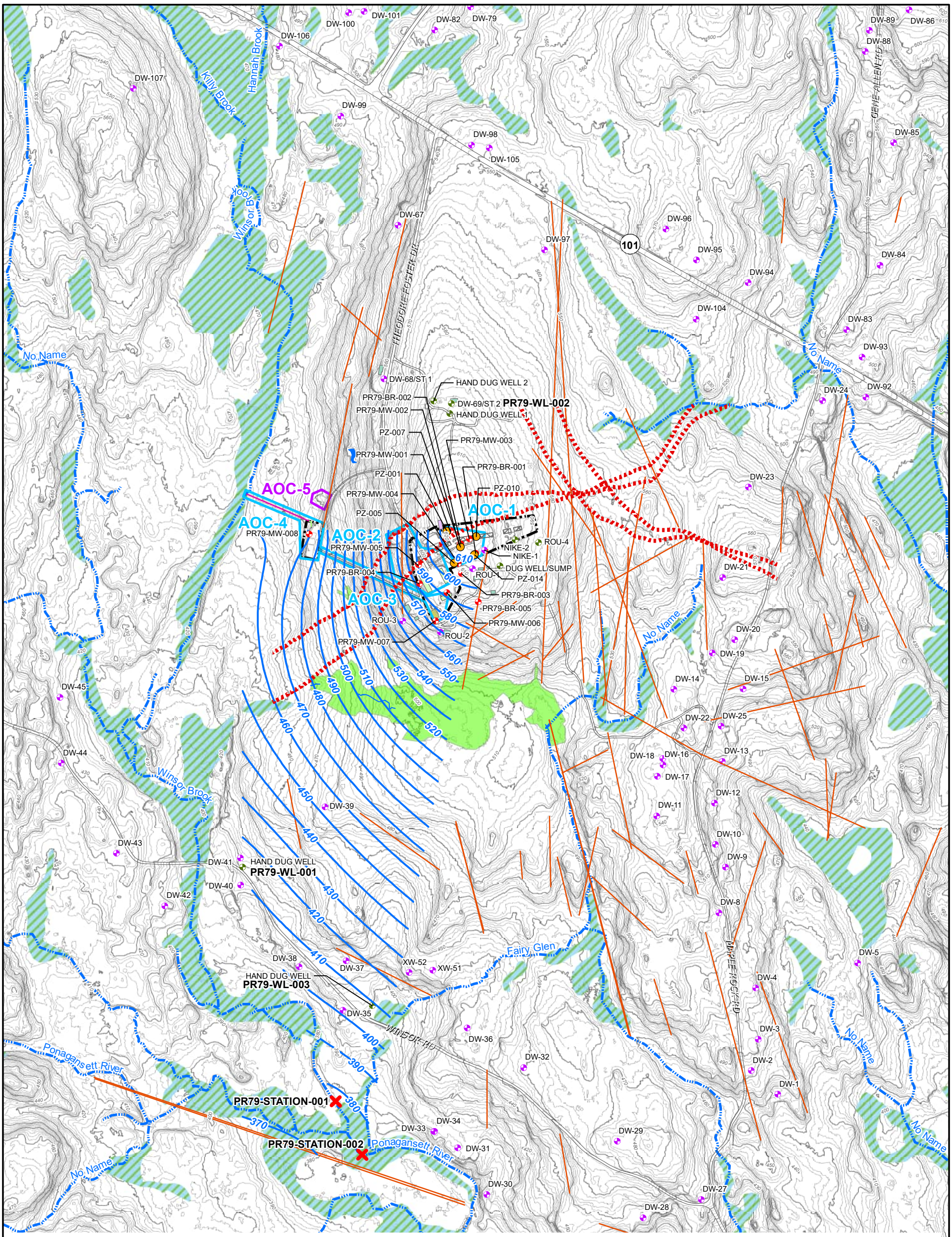
QAPP Worksheet #28-7: Analytical Quality Control and Corrective Action – Metals and SPLP (Mercury)

Matrix	Aqueous and Solids
Analytical Group	Metals, SPLP Metals (Mercury)
Analytical Method/ SOP Reference	SW-846 7470A/7471B/ CA-611, CA-615

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	AQ: 82-119 %R SL: 80-124	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch.	Same as LCS RPD of all analytes ≤ 20% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results between DL and LOQ. Acceptance range for all metals LOQ verification is 67-133%R.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

MB – method blank

Figures



Legend

- Gauging Station
- Bedrock Monitoring Well
- Drinking Water - Individual
- Drinking Water - Not in Use
- Stream
- Perimeter Fence
- Bedrock Lineament
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Area of Concern (AOC) (Non-Government)
- Wetland (Woodard & Curran, 2019)
- Wetlands (RIGIS 1993)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Index Contour
- Intermediate Contour
- Sewer Line
- Drainage
- Groundwater Contour
- Fractured Bedrock
- Fracture - Ground Surface Intersects

AECOM

Drawn:	JB	11/21/2022
Approved:	KK	11/21/2022
Project #:	60608703	

Sources
Structures, Utilities, Contours (The Johnson Company, 2018)
Surface Water Bodies (The Johnson Company, 2018)
Wetlands (RIGIS 1993) (Woodard & Curran, 2019)
Historical Feature/Activity Locations (USAGC, 2015)
Bedrock Lineaments (The Johnson Company, 2018)

N

0 375 750
Scale in Feet

FIGURE 10-2
WELL LOCATION MAP

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: L:\Legacy\USCHL1FP001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO W912WJ19F0110 NIKE PR79 RIFS\900_GIS\Projects\QAPP_Addendum_202209\MXD\Fig_10_2_Well_Location_Map.mxd

PR79-SB-141/PR79-MW-009
PR79-SB-142/PR79-MW-010
PR79-SB-101/PR79-MW-001
PZ-011
PZ-001
PR 79 C RM 2
PR 79 C 2 RM 4
FORMER RADAR PAD B (Demolished)
FORMER 6,000 GALLON DIESEL FUEL UST (Removed)
FORMER BATTERY CONTROL VAN (Demolished)
CESSPOOL 1
CESSPOOL 2
APPROXIMATE LOCATION OF DUMPING
PR79-SB-105
ANTENNA POLE (NIC)
GPR 5
PZ-009
PZ-010
FORMER INTERCONNECTING CORRIDOR (Demolished)
PZ-002
SB-008
PR79-SB-104/PR79-MW-003
PZ-019
SB-020
PR79-SB-120
PR 79 C
FORMER RADAR CONTROL VAN (Abandoned)
FORMER RADAR PAD C (Demolished)
FORMER FREQUENCY CHANGER/GENERATOR BUILDING (Demolished)
FLOOR DRAIN
PR79-BR-002
AOC-1
PR 79 C RM 1
PR 79 C 2 RM 5
PR79-SB-103/PR79-MW-002
PZ-004
PR 79 C 2
PZ-003
PR79-SB-102
PZ-007
DRY WELL
NIKE-2
NIKE-1
PZ-022
FORMER TRANSFORMER PADS
FORMER WATER STORAGE TANK (Demolished)
GPR 4
GPR 3
GPR 2
AOC-2
AS-BUILT 16-06-109 (JUNE 1957)
HELI PAD
PR79-SB-107/PR79-BR-001
PZ-014
PZ-006
FORMER RADAR PAD A (Demolished)
PR 79 C RM 3
PZ-005
PR79-SB-106/PR79-MW-004
FORMER WATER PUMP HOUSE (Demolished)
SHED (in use)
DUG WELL/SUMP
ROU-1
BARN (in use)
620
610
600
590
618
614
612
608
606
604
602
598
596
594

Groundwater Contour
Bedrock Lineament
Fractured Bedrock
Fracture - Ground Surface Intersects

Sources
Orthoimagery (RIGIS, 04/2022)
Structures, Utilities, Contours (The Johnson Company, 2018)
Surface Water Bodies (The Johnson Company, 2018)
Historical Feature/Activity Locations (USAGC, 2015)

AECOM

Drawn:	JB	11/21/2022
Approved:	KK	11/21/2022
Project #:	60608703	

Legend

- Proposed Boring/Well Location
- Monitoring Well
- Soil Boring
- Historic Soil Boring/Piezometer (AMEC, 2013)
- Abandoned Historic Soil Boring/ Piezometer (AMEC, 2013)
- Historic Soil Boring (AMEC, 2013)
- Drinking Water - Individual
- Drinking Water - Not in Use
- Geodetic Control Station (National Geodetic Survey)
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Cemetery
- Index Contour
- Intermediate Contour
- Electrical Line
- Sewer Line
- Water Line
- Drainage
- Seismic Lines (Hager Geosciences, Inc (HGI), 2017)
- Groundwater Contour

0 25 50
Scale in Feet

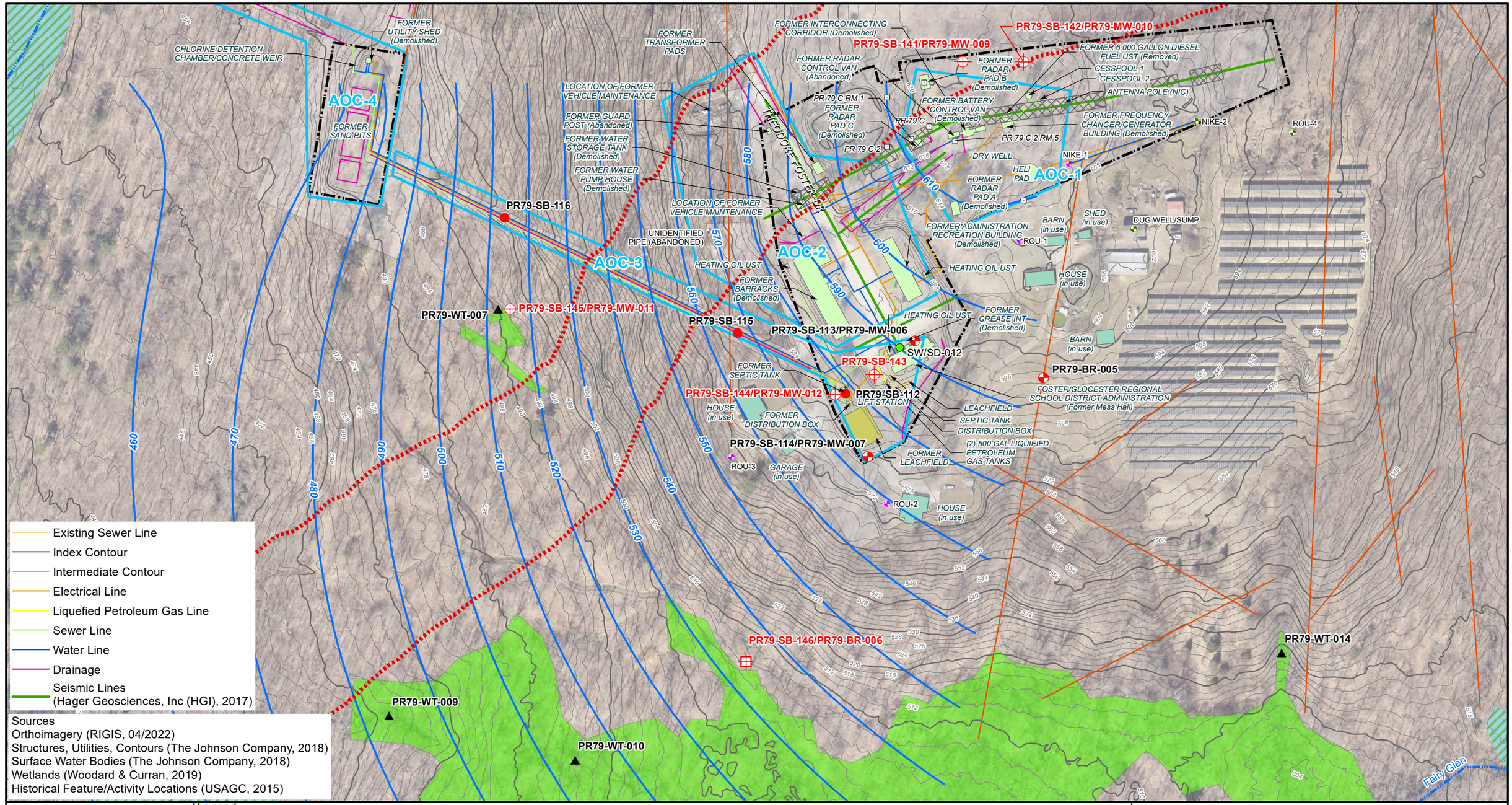
N

FIGURE 17-1A
AOC-1 SAMPLING PLAN

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: L:\Legacy\USCHL1FP001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO W912WJ19F0110 NIKE PR79 RIFS\900_GIS\Projects\QAPP_Addendum_202209\MXD\Fig_17_1A_AOC_1_Sampling_Plan.mxd



AECOM

Drawn:	JB	11/21/2022
Approved:	KK	11/21/2022
Project #:	60608703	

Legend

- Proposed Boring/Bedrock Well Location
- Proposed Boring/Well Location
- Monitoring Well
- Soil Boring
- Historic Soil Boring (AMEC, 2013)
- Surface Water/Porewater/Sediment
- Drinking Water - Individual
- Drinking Water - Not in Use
- Unidentified Pipe
- Geodetic Control Station (National Geodetic Survey)
- Stream
- Perimeter Fence
- Former Government Property
- Non-Government Property
- Area of Concern (AOC)
- Wetland (Woodard & Curran, 2019)
- Extent of Former NIKE PR-79 Control Area (USAGC, 2015)
- Former Sewer and Stormwater Structures
- Existing Sewer Structures
- Bedrock Lineament
- Groundwater Contour
- Fractured Bedrock
- Fracture - Ground Surface Intersects

N

0 90 180

Scale in Feet

FIGURE 17-1B
AOC-3 SAMPLING PLAN

DRAFT

FORMER NIKE PR-79 CONTROL AREA
FOSTER, RHODE ISLAND

Path: L:\Legacy\USCHL1FP001\Data\Projects\Govt\Projects\USACE New England HTRW 2019\Task Orders\TO W912WJ19F0110 NIKE PR79 RIFS\900_GIS\Projects\QAPP_Addendum_202209\MXD\Fig_17_1B_AOC_3_Sampling_Plan.mxd

Appendix A: Updated AECOM Field SOPs

Utility Clearance

Procedure 3-01

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.

1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

1.5 If there are procedures, whether it be from AECOM, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific Quality Assurance Project Plan (QAPP).

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

3.0 Terms and Definitions

3.1 **Utility**

For the proposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

3.2 **As-Built Plans**

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

3.3 **One-Call**

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at www.call811.com.

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type or work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

3.4 **Toning**

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

4.0 Training and Qualifications

4.1 The **Task Order (TO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Equipment and Supplies

5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

6.0 Procedure

Proceed with the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.

6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific QAPP. Include as many of the cultural and natural features as practical in this plan.

6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the TO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The TO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a TO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

6.3 **Site Visit/Locate Utilities/Toning**

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radio detection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.
- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed

exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the client, the TO Manager, and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the client to verify its accuracy prior to initiating subsurface sampling activities.

7.0 Quality Control and Assurance

7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.

8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

9.0 Attachments or References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual.* Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist	Bob Shoemaker Senior Scientist	Rev 0 – Initial Issue (June 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley Environmental Scientist	Richard Purdy Project Scientist	Rev 2 – Update & Review (June 2022)

Investigation Derived Waste Management

Procedure 3-05

1.0 Purpose and Scope

This standard operating procedure (SOP) describes activities and responsibilities of the client with regard to management of investigation-derived waste (IDW). The purpose of this procedure is to provide guidance for the minimization, handling, labelling, temporary storage, inventory, classification, and disposal of IDW generated under the client contract. This procedure will also apply to personal protective equipment (PPE), sampling equipment, decontamination fluids, non-IDW trash, non-indigenous IDW, and hazardous waste generated during implementation of removal or remedial actions. The information presented will be used to prepare and implement work plans (WPs) for IDW-related field activities. The results from implementation of WPs will then be used to develop and implement final IDW disposal plans.

If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to IDW then those procedures may be added as an appendix to the project specific SAP.

This procedure shall serve as management-approved professional guidance for the client and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by both the Task Order (TO) Manager and the Quality Assurance (QA) Manager or Technical Director and documented.

This procedure was developed to serve as management-approved professional guidance for the management of IDW generated under the client contract. It focuses on the requirements for minimizing, segregating, handling, labelling, storing, and inventorying IDW in the field. Certain drum inventory requirements related to the screening, sampling, classification, and disposal of IDW are also noted in this procedure.

2.0 Safety

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the TO WP and/or direction from the **Site Safety Officer (SSO)**.

All **Field Personnel** responsible for IDW management must adhere to the HASP and must wear the PPE specified in the site-specific HASP. Generally, this includes, at a minimum, steel-toed boots or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). If safe alternatives are not achievable, discontinue site activities immediately.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **TO Manager** is responsible for ensuring that IDW management activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in IDW management shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all IDW is managed according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

All AECOM personnel who will perform any duties related to management of Resource Conservation and Recovery Act (RCRA) hazardous wastes or shipping of Department of Transportation (DOT) Hazardous Materials will be properly trained in accordance with 40 CFR § 262.34 and §265.16 for RCRA Waste Generators, as well as 49 CFR § 172.704 for DOT Hazardous Materials Shippers. All RCRA Hazardous Wastes are by definition DOT Hazardous Materials. See Section 6.1 for details on determining the IDW waste classification.

5.0 Equipment and Supplies

The equipment and supplies required for implementation of this SOP include the following:

- Containers for waste (e.g., [U.S. Department of Transportation] DOT approved 55-gallon open and closed top drums) and material to cover waste to protect from weather (e.g., plastic covering);
- Hazardous /non-hazardous waste drum labels (weatherproof);
- Permanent marking pens;
- Inventory forms for project file;
- Plastic garbage bags, zip lock storage bags, roll of plastic sheeting; and
- Steel-toed boots, chemical resistant gloves, coveralls, safety glasses, and any other PPE required in the HASP.

6.0 Procedure

The following procedures are used to handle the IDW.

6.1 **Drum Handling**

6.1.1 IDW shall be containerized using DOT approved drums. The drums shall be made of steel or polyethylene, be completely painted or opaque, and have removable lids (i.e., United Nations Code 1A2 or 1H2). Always consider IDW physical and chemical characterises to make sure the drum material is compatible. Typically, 55-gallon drums are used, however small drums may be used depending on the amount of waste generated. Large overpack drums may be used if smaller drums become damaged. New drums are preferred. The use of recycled drums should be avoided.

6.1.2 Recycled drums should not be used for hazardous waste, PCBs or other regulated shipments. For short-term storage of liquid IDW prior to discharge, double-walled bulk steel or plastic storage tanks may be used. For this scenario, consider the scheduling and cost-effectiveness of this type of bulk storage, treatment, and discharge system versus longer-term drum storage.

6.1.3 For long-term IDW storage at other project locations, the DOT approved drums with removable lids are recommended. Verify the integrity of the foam or rubber sealing ring located on the underside of some drum lids prior to sealing drums containing IDW liquids.

6.1.4 If the sealing ring is only partially attached to the drum lid, or if a portion of the sealing ring is missing, select another drum lid with a sealing ring that is in sound condition.

6.1.5 To prevent damage to drums, loss of drum integrity/containment, and/or presenting hazards to drum handlers, the following "Rules-of-Thumb" should be applied when filling drums.

- Liquid, soil, PPE/plastics, and construction debris must be segregated by media into individual drums.
- A **void space of 4 to 6 inches** from the top of the drum (the upper drum ring on most drums) will be left in the drum to allow room for ice expansion when filling drums with water or oil/water emulsions. Under freezing temperatures, expanding ice in a full drum can deform the bottom of a drum such that it is no longer DOT compliant, cause ruptures and/or dislodge the drum lid and present a containment breach. The consequences of this damage can be both economic and environmental.
- Compatibility between the chemical component(s) of the IDW and the drum material must be considered before choosing the type of drum/container to use. Steel drums are susceptible to corrosion and loss of integrity when in contact with high pH water. Lime-based products (cement, concrete, grout, etc.) should not be disposed in steel drums containing water or soil water mixtures, and liquid IDW should not be disposed in steel drums used to mix lime-based products (separate reusable containers for mixing should be used when possible). If high (>12) or low (<2) pH conditions are possible, IDW liquids should be monitored for pH using a calibrated pH meter or pH test strips. The use of plastic drum liners or polyethylene drums is also recommended for high or low pH liquid IDW.
- Soil drums will be filled to no more than two thirds of the drum capacity. Drums completely full of soil can weigh over 600 pounds. Although drum handling tools and carts provide some assistance, moving such excessive weights present significant hazards, including; muscle strain, crushing (foot and fingers), and loss of drum control, such as sliding off of lift gates.
- Drums should not be overfilled filled with PPE and plastic (tubing, old macrocores) such that the material is excessively compacted. Pinch points are presented as the drum is closed under force, and the compressed material can spring up when the drums are opened.

6.1.6 Stacking full or partially full drums is prohibited.

6.1.7 To prepare IDW drums for labelling, wipe clean the outer wall surfaces and drum lids of all material that might prevent legible and permanent labelling. If potentially contaminated material adheres to the outer surface of a drum, wipe that material from the drum, and segregate the paper towel or rag used to remove the material with visibly soiled PPE and disposable sampling equipment. Label all IDW drums and place them on pallets prior to storage.

6.2 **Labelling**

6.2.1 Containers used to store IDW must be properly labelled. Two general conditions exist: 1) from previous studies or on-site data, waste characteristics are known to be either hazardous or nonhazardous; or 2) waste characteristics are unknown until additional data are obtained.

6.2.2 For situations where the waste characteristics are known, the waste containers should be packaged and labelled in accordance with state regulations and any federal regulations that may govern the labelling of waste.

6.2.3 The following information shall be placed on all non-hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.4 The following information shall be placed on all hazardous waste labels:

- Description of waste (i.e., purge water, soil cuttings);
- Generator information (i.e., name, address, contact telephone number);
- EPA identification number (supplied by on-site client representative);
- Date when the waste was first accumulated.

6.2.5 When the final characterization of a waste is unknown, a notification label should be placed on the drum with the words “waste characterization pending analysis” and the following information included on the label:

- Description of waste (i.e., purge water, soil cuttings);
- Contact information (i.e., contact name and telephone number);
- Date when the waste was first accumulated.

6.2.6 Once the waste has been characterized, the label should be changed as appropriate for a nonhazardous or hazardous waste.

6.2.7 Waste labels should be constructed of a weatherproof material and filled out with a permanent marker to prevent being washed off or becoming faded by sunlight (faded entries should be remarked during inspections performed as specified in Section 6.2.4). It is recommended that waste labels be placed on the side of the container, since the top is more subject to weathering. However, when multiple containers are accumulated together, it may also be helpful to include labels on the top of the containers to facilitate organization and disposal. In addition to a label, each drum should be numbered on the side and top with a paint pen or wax pencil for easy identification.

6.2.8 Each container of waste generated shall be recorded in the field notebook used by the person responsible for labelling the waste. After the waste is disposed of, either by transportation off-site or disposal on-site in an approved disposal area, an appropriate record shall be made in the same field notebook to document proper disposition of IDW.

6.3 Types of Site Investigation Waste

Several types of waste are generated during site investigations that may require special handling. These include solid, liquid, and used PPE, as discussed further below.

Solid Waste

Soil cuttings from boreholes will typically be placed in containers unless site specific requirements allow for soil cuttings to be placed back into the borehole after drilling is complete. Drilling mud generated during investigation activities shall be collected in containers. Covers should be included on the containers and must be secured at all times and only open during filling activities. The containers shall be labelled in accordance with this SOP. An inventory containing the source, volume, and description of material put in the containers shall be logged on prescribed forms and kept in the project file.

Non-hazardous solid waste can be disposed on-site in the designated site landfill or in a designated evaporation pond if it is liquefied. Hazardous wastes must be disposed off-site at an approved hazardous waste landfill.

Liquid Waste

Groundwater generated during monitoring well development, purging, and sampling can be collected in truck-mounted containers and/or other transportable containers (i.e., 55-gallon drums). Lids or bungs on drums must be secured at all times and only open during filling or pumping activities. The containers shall be labelled in accordance with this SOP. Non-hazardous liquid waste can be disposed of in one of the designated lined evaporation ponds on-site. Hazardous wastes must be handled separately and disposed off-site at an approved hazardous waste facility.

Personal Protective Equipment

PPE that is generated throughout investigation activities shall be placed in plastic garbage bags. If the solid or liquid waste that was being handled is characterized as hazardous waste, then the corresponding PPE should also be disposed as hazardous waste. If not, all PPE should be disposed as non-hazardous waste in the designated on-site landfill. Trash that is generated as part of field activities may be disposed of in the landfill as long as the trash was not exposed to hazardous media.

6.1 IDW Waste Classification

State and federal regulations require specific handling and storage requirements for wastes classified as hazardous, such as secondary containment and waste removal deadlines (see Section 6.2.2). The Site owner/operator must determine whether the IDW may contain a listed hazardous waste based on the source of contamination, contaminants, and waste manifests or any other documentation of wastes generated at the Site. It is presumed that the IDW will be considered a solid waste (40 CFR 261.2) but this should be verified during the work plan development. If the available documentation indicates that a listed hazardous waste was generated at the Site, then the IDW will be considered a hazardous waste regulated under RCRA.

If there is inconclusive documentation concerning the IDW generated at the Site, then the U.S. EPA has stated the IDW is not a listed hazardous waste. However, in this case, further evaluation is necessary to evaluate whether the IDW in question exhibits a characteristic of hazardous waste. This is determined by analytical testing or knowledge. An IDW that may be characteristically hazardous should be evaluated for the following hazardous characteristics:

- Characteristic of ignitability (40 CFR §261.21)
- Characteristic of corrosivity (40 CFR §261.22)
- Characteristic of reactivity (40 CFR §261.23)
- Characteristic of toxicity (40 CFR §261.24)

If the RDW contains a listed hazardous waste, then U.S. EPA's contained-in policy (53 FR 31138, 31142, 31148, 57 FR 21453, 61 FR 18795) for contaminated environmental media should be evaluated. U.S. EPA considers IDW to contain hazardous waste:

- when it exhibits a characteristic of hazardous waste; or
- when it is impacted with concentrations of hazardous constituents from listed hazardous wastes that are above health-based levels.

Generally, IDW that does not (or no longer) contain hazardous waste are not subject to RCRA, but in some circumstances, the IDW that contained hazardous waste when first generated remain subject to land disposal restrictions (LDR) (40 CFR §268.45). There are also special LDR standards specific to contaminated debris (40 CFR §268.45).

6.2 **Waste Accumulation On-Site**

6.2.1 Solid, liquid, or PPE waste generated during investigation activities that are classified as nonhazardous or "characterization pending analysis" should be disposed of as soon as possible. Until off-site transport and disposal is arranged, drums should be moved to a staging location accessible by pickup by truck. This location should be relatively flat, have a hard surface (densely compact dirt, concrete, or asphalt), and be secure (by a fence or building).

6.2.2 Solid, liquid, or PPE waste generated during investigation activities that are classified as hazardous **shall not** be accumulated on-site longer than **90 days**. All hazardous waste containers shall be stored in a secured storage area. The following requirements for the hazardous waste storage area must be implemented:

- Proper hazardous waste signs shall be posted as required by any state or federal statutes that may govern the labelling of waste;
- Secondary containment to contain spills;
- Spill containment equipment must be available;
- Fire extinguisher;
- Adequate aisle space for unobstructed movement of personnel.

6.2.3 When possible, drums should be segregated in the storage area by media and or classification (liquid, solid, non-hazardous, hazardous, etc.) to facilitate type identification during characterization sampling and pickup and reduce the need to rearrange drums if multiple pickups by type are required.

6.2.4 Throughout the project, an inventory shall be maintained to itemize the type and quantity of the waste generated. During active site work, weekly storage area inspections should be performed and documented to ensure compliance with the requirements specified above. Monthly storage area inspections should be performed following the completion of active site work and the date the IDW is removed from the storage area by the waste hauler. Containers should be inventoried and inspected regularly. Labels should be checked to make sure they remain legible. Inspection notes should include the condition of the staging area as this will be important when coordinating the labour and equipment the waste hauler will require. Anomalies should be documented and photographed.

6.3 **Waste Disposal**

6.3.1 Solid, liquid, and PPE waste will be characterized for disposal through the use of client knowledge, laboratory analytical data created from soil or groundwater samples gathered during the field activities, and/or composite samples from individual containers. The selected disposal facility will prepare a waste profile based on the characterization results. The waste generator (Navy representative or authorized agent) will review and sign the profile.

6.3.2 All waste generated during field activities will be stored, transported, and disposed of according to applicable state, federal, and local regulations. All wastes classified as hazardous will be disposed of at a licensed treatment storage and disposal facility or managed in other approved manners.

6.3.3 Disposal facilities for waste generated during activities under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) will require EPA approval under the Off-Site Rule (OSR) procedures (40 CFR 300.440) to ensure the facility is operating in compliance with RCRA or other Federal and State requirements. After the

waste profile is finalized, the generator will submit it with an OSR request form to the EPA project manager for approval. An example OSR request form is provided in Attachment A. IDW may not be shipped to the facility until approval is granted by the EPA. OSR approvals per waste profile are valid for 90 days.

6.3.4 In general, waste disposal should be carefully coordinated with the facility receiving the waste. Facilities receiving waste have specific requirements that vary even for non-hazardous waste, so characterization should be conducted to support both applicable regulations and facility requirements.

6.4 **Regulatory Requirements**

The following federal and state regulations shall be used as resources for determining waste characteristics and requirements for waste storage, transportation, and disposal:

- Code of Federal Regulations (CFR), Title 40, Part 261; and
- CFR, Title 49, Parts 172, 173, 178, and 179.

6.5 **Waste Transport**

A state-certified hazardous waste hauler shall transport all wastes classified as hazardous. Typically, the facility receiving any waste can coordinate a hauler to transport the waste. Shipped hazardous waste shall be disposed of in accordance with all RCRA/USEPA requirements. All waste manifests or bills of lading will be signed either by the client or the client's designee.

7.0 Quality Control and Assurance

7.1 Management of IDW must incorporate quality control measures to ensure conformance to these and the project requirements.

8.0 Records, Data Analysis, Calculations

8.1 Maintain records as required by implanting the procedures in this SOP.

8.2 Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

9.0 Attachments or References

Department of Defence, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of Energy, United States (DOE). 1994. *The Off-Site Rule*. EH-231-020/0194. Office of Environmental Guidance. March.

1999. *Management of Remediation Waste under the Resource Conservation and Recovery Act (RCRA)*. Office of Environmental Policy and Assistance. 20 December.

Environmental Protection Agency, United States (EPA). 1991. *Management of Investigative-Derived Wastes During Site Inspections*. Office of Emergency and Remedial Response. EPA/540/G-91/009. May.

1992a. *Guidance for Performing Site Inspections under CERCLA*. EPA/540/R-92/021. Office of Emergency and Remedial Response. September.

1992b. *Guide to Management of Investigative-Derived Wastes*. Quick reference fact sheet. OSWER Dir. 9345.3-03FS. Office of Solid Waste and Emergency Response. January.

1997a. *Sending Wastes Off Site? OSC and RPM Responsibilities under the Off-Site Rule*. EPA/540-F-97-006, Office of Solid Waste and Emergency Response. September.

1997b. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Final Update IIIA. Office of Solid Waste. Updates available: www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

1998. *Management of Remediation Waste under RCRA*. EPA/530-F-98-026. Office of Solid Waste and Emergency Response. October.

(No Date). *Compliance with the Off-Site Rule During Removal Actions.* Office of Regional Counsel (Region 3). Hendershot, Michael.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Joshua Millard Senior Geologist	Andrew Borden Geologist	Rev 1 – Technical (Jan 2017)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 2 – PFAS sampling update (July 2019)
Rose Kelley Environmental Scientist	Richard Purdy Project Scientist	Rev 3 – Update & Review (June 2022)

Attachment A
Off Site Rule Request Form

United States Environmental Protection Agency – Region 1

Off-Site Rule Compliance Request Form

Date: (mm/dd/yy)		Supporting Documentation Required-Attached? (yes/no)	

RECEIVING FACILITY INFORMATION:		
1	**Name of Facility receiving CERCLA waste:**	
2	**Address of Facility:**	
3	**City:**	
4	**State:**	
5	**Zip Code:**	
6	**EPA/State Facility ID:**(e.g. Haz. Waste/Municipal Waste ID)	
7	**Other Pertinent ID Numbers:** (e.g. License #, permit #)	
8	**Phone Number** (if available):	
9	**Contact Name** (if available):	
10	**FAX Number** (if available):	
11	**E-mail address** (if available):	

GENERATING FACILITY INFORMATION:		
12	**CERCLA Site Name:**	
13	**CERCLA Site Address:**	
14	**City:**	
15	**State:**	
16	**Zip Code:**	
17	**CERCLA Site ID:** (i.e. alpha-numeric)	
18	**EPA CERCLA ID #:**	
19	**Waste Media:** (e.g., Soil, Water, Air, etc.)	
20	**CERCLA Hazardous Waste Contaminates:** (e.g. tce, lead)	
21	**Amount of CERCLA Waste:** (e.g. gallons, pounds, tons, ft^3, yd^3)	
22	**EPA representative making waste determination:** (e.g. OSC, RPM & Tel.#)	
23	**Basis of Waste Determination:** (e.g. analyses, TCLP, etc.)	

[Form: Off-Site Compliance Request] [Rev. G – August 25, 2016] [MacLeod.Donald@epa.gov]

For more information on the Off-Site Rule, please contact the appropriate Regional Off-Site Contact (ROC) listed at **http://www.epa.gov/waste/hazard/wastetypes/wasteid/offsite/index.htm**

	Regional Off-Site Contacts (listed as of April 8, 2014)	
Region # U.S. & DC,PR,VI	**Contact Name**	**Telephone #**
1 CT,MA,ME,NH,RI,VT	Donald MacLeod (macleod.donald@epa.gov)	**617.918.1405**
2 NY,NJ,PR,VI	Beckett Grealish (Region2_OSR@epa.gov)	**732.321.4341**
3 DC,DE,MD,PA,VA,WV	Stacie Pratt (pratt.stacie@epa.gov)	**215.814.5173**
4 AL,FL,GA,KY,MS,NC,SC,TN	Paula Whiting (whiting.paula@epa.gov)	**404.562.9277**
5 IL,IN,MI,MN,OH,WI	William Damico (damico.william@epa.gov)	**312.353.8207**
6 AR,LA,NM,OK,TX	Wilkin (Ron) Shannon (shannon.wilkin@epa.gov)	**214.665.2282**
7 IA,KS,MO,NE	Nicole Moran (moran.nicole@epa.gov)	**913.551.7641**
8 CO,MT,ND,SD,UT,WY	Linda Jacobson (jacobson.linda@epa.gov)	**303.312.6503**
9 AZ,CA,HI,NV	Kandice Bellamy (bellamy.kandice@epa.gov)	**415.972.3304**
10 AK,ID,OR,WA	Kevin Schanilec (schanilec.kevin@epa.gov) Ofelia Erickson (erickson.ofelia@epa.gov)	**206.553.1061** **206.553.2583**

Equipment Decontamination

Procedure 3-06

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.

1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

It is the responsibility of the **Site Safety and Health Officer (SSHO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally, the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Task Order (TO) Quality Assurance Project Plan (QAPP) and deionized water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP) and must wear the personal protective equipment (PPE) specified in the site-specific APP/SSHP. Generally, this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSHO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

2.1 **Chemical Hazards associated with Equipment Decontamination**

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water;
- Utilize PPE as specified in the site-specific APP/SSHP to maximize splash protection;
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory); and
- Take the necessary precautions when handling detergents and reagents.

2.2 **Physical Hazards associated with Equipment Decontamination**

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level;
- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel; and

- Take necessary precautions when handling field sampling equipment.

3.0 Terms and Definitions

None.

4.0 Training and Qualifications

4.1 The **TO Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.

4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.

4.4 All **Field Personnel** are responsible for the implementation of this procedure.

5.0 Procedure

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted;
- Types of equipment requiring decontamination;
- Frequency of equipment decontamination;
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern;
- Method for containing the residual contaminants and wash water from the decontamination process; and
- Use of a quality control measure to determine the effectiveness of the decontamination procedure.

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

5.1 **Decontamination Area**

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

5.2 **Types of Equipment**

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and

caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

5.3 Frequency of Equipment Decontamination

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

5.4 Cleaning Solutions and Techniques

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a PFAS-free detergent (Alconox®, Liquinox®, or other suitable detergent) and deionized water solution, and (2) rinse in triplicate with deionized water. If possible, disassemble equipment prior to cleaning. Add an additional wash as needed at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and deionized water solution, and (2) rinse in triplicate with deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analysed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of deionized water. Decontamination solvents to be used during field activities will be specified in the TO QAPP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized water after each measurement. Also wash new, unused soil sample liners and caps with a fresh detergent solution and rinse them with deionized water to remove any dirt or cutting oils that might be on them prior to use.

5.5 Containment of Residual Contaminants and Cleaning Solutions

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

6.0 Quality Control and Assurance

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free deionized water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a PFAS-free cotton cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

7.0 Records, Data Analysis, Calculations

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

8.0 Attachments or References

8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

8.2 Procedure 3-05, *IDW Management*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley Environmental Scientist	Richard Purdy Project Scientist	Rev 2 – Update & Review (June 2022)

AECOM

Monitoring Well Development

Procedure 3-13

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.

1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:

- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
- Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process; and
- Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.

1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the Task Order (TO) Manager and other stakeholders, as applicable.

1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Quality Assurance Project Plan (QAPP). For project-specific information refer to the QAPP, which takes precedence over these procedures.

1.5 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP). Work will be conducted according to the TO QAPP and/or direction from the Site Safety and Health Officer (SSHO).

2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.

4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.

4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.

4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The TO Manager is responsible for ensuring that well development activities comply with this procedure. The TO Manager is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Site Supervisor (SS) is responsible for ensuring that all well development activities are conducted according to the either this procedure or the applicable procedure presented in the project-specific QAPP.

5.2.4 Field sampling personnel are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.

6.2 Equipment and materials used for well development may include, but is not limited to:

Well development equipment

- Surge block
- Disposable HDPE bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells

- Watterra® footvalve
- PFAS-free (e.g., Teflon-free) electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, potential of hydrogen (pH), conductivity, dissolved oxygen (DO), and oxidation reduction potential (ORP)
- Instrument calibration solutions
- Teflon-free water level meter
- Oil/water interface probe

General equipment

- Project-specific plans including the site-specific APP/SSHP and QAPP
- Non-water-repellent field notebook/field forms/site maps
- Ball point pens or fine-point indelible marker
- 5-gallon HDPE or polypropylene buckets

Equipment decontamination supplies (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE) as specified by the APP/SSHP
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- PFAS-free deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

7.0 Procedure

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

7.1 General Preparation

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.

- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a Teflon-free water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra® pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific QAPP, or State or local requirements. At this point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific QAPP are met.

7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific QAPP.
- In some cases, the bailer or Watterra® foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen and pushing water out into the formation to break down bridging.

- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific QAPP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific QAPP.
- The lifting and lowering action of the Watterra® system will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.
- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific QAPP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

7.3 Discharge Monitoring

7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged discharge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific QAPP. Water quality parameters should be measured on each well volume removed.

7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific QAPP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.
- Groundwater parameters for three consecutive standing water volumes are within the following:
  - pH – within ± 0.2 units
  - Specific conductivity – within ± 3%
  - ORP – within ± 10 mV
  - Temperature – within ±1 degree Celsius
  - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific QAPP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific QAPP.

7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that

exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.

7.6 Temporary Well Points

For certain projects, temporary well points (TWPs) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with HDPE tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific QAPP.

8.0 Quality Control and Assurance

8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific QAPP.

8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific QAPP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

9.0 Records, Data Analysis, Calculations

9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:

- Well Location;
- Weather conditions;
- Date and Time;
- Purge Method; and
- Reading/measurements obtained.

10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

Author	Reviewer	Revisions (Technical or Editorial)
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley, Environmental Scientist	Richard Purdy, Project Scientist	Rev 2 – Update & Review (June 2022)

Attachment 1
Well Development Record

Well/Piezometer Development Record

Well ID:

Client: _____

Project No: _____ Date: _____ Developer: _____

Site Location: _____

Well/Piezometer Data

Well ☐ Piezometer ☐ Diameter _____ Material _____

Measuring Point Description _____ Geology at Screen Interval (if known) _____

Depth to Top of Screen (ft.) _____

Depth to Bottom of Screen (ft.) _____ Time of Water Level Measurement _____

Total Well Depth (ft.) _____ Calculate Purge Volume (gal.) _____

Depth to Static Water Level (ft.) _____ Disposal Method _____

Headspace _____

Original Well Development ☐ Redevelopment ☐ Date of Original Development _____

DEVELOPMENT METHOD _____

PURGE METHOD _____

Time	Total Volume Purged (gal.)	Flow Rate (gpm)	Turbidity (NTU)	Color	pH	Temp	Other

ACCEPTANCE CRITERIA (from workplan)

Minimum Purge Volume Required _____ gallons
Maximum Turbidity Allowed _____ NTUs
Stabilization of parameters _____%

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Has parameters stabilized	☐	☐	☐

If no or N/A explain below:

_____

_____

_____

Signature _____ Date: _____

Monitoring Well Sampling

Procedure 3-14

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the actions to be used during monitoring well sampling activities and establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples that are representative of aquifer conditions with as little alteration to water chemistry as possible.

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. All field sampling personnel responsible for sampling activities must review the project-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP) paying particular attention to the control measures planned for the well sampling tasks. Conduct preliminary area monitoring of sampling wells to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and liquid matrix through the use of appropriate personal protective equipment (PPE).

2.2 Observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves and rubberized steel-toed boots. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on evaluation for PFAS and on the contaminant concentrations. Refer to the project-specific APP/SSHP for the required PPE.

2.3 Physical Hazards associated with Well Sampling

- To avoid lifting injuries associated with pump and bailers retrieval, use the large muscles of the legs, not the back.
- Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
- When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid slip/trip/fall conditions as a result of pump discharge, use textured boots/boot cover bottoms.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility due to PPE.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross-contamination will occur through the following:

- The use of clean sampling tools at each location as necessary; and
- Avoidance of material that is not representative of the media to be sampled.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **Task Order (TO) Manager** is responsible for ensuring that monitoring well sampling activities comply with this procedure. The **TO Manager** is responsible for ensuring that all field sampling personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

6.0 Equipment and Supplies

6.1 Purging and Sampling Equipment

- Pump (Peristaltic, Portable Bladder, Submersible)
- Polyethylene bladders (for portable bladder pumps)
- Bladder pump controller (for portable bladder pumps)
- Air compressor (for portable bladder pumps)
- Nitrogen cylinders (for portable bladder pumps)
- 12-volt power source
- Polyethylene inlet and discharge tubing
- Silicone tubing appropriate for peristaltic pump head
- HDPE bailer appropriately sized for well
- Disposable bailer string (polypropylene)
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Turbidity meter
- Teflon-free water level meter
- Oil/water interface probe

6.2 General Equipment

- Sample kit (i.e., bottles, labels, preservatives, custody records and tape, cooler, wet ice)
- Sample Chain-of-Custody (COC) forms
- Sample Collection Records
- Sample packaging and shipping supplies
- Fine-tipped Sharpie® marker
- Deionized water supply
- Polyethylene water dispenser bottles
- HDPE flow measurement cup or bucket
- 5-gallon buckets
- Instrument calibration solutions
- Stopwatch or watch
- Disposable, powderless Nitrile gloves
- Cotton towels
- Trash bags
- Zipper-lock (e.g., Ziploc brand) bags
- Equipment decontamination supplies (e.g., Alconox®, Liquinox®, NOT Decon 90™)
- Health and safety supplies (as required by the APP/SSHP)
- Approved plans such as: project-specific APP/SSHP and Quality Assurance Project Plan (QAPP)
- Well keys or combinations
- Monitoring well location map(s)
- Field project logbook/ballpoint pen

7.0 Calibration or Standardization

7.1 Field instruments will be calibrated daily according to the requirements of the QAPP and manufacturer's specifications for each piece of equipment. Equipment will be checked daily with the calibration solutions at the end of use of the equipment. Calibration records shall be recorded in the field logbook or appropriate field form.

7.2 If readings are suspected to be inaccurate, the equipment shall be checked with the calibration solutions and/or re-calibrated.

8.0 Procedure

8.1 Preparation

8.1.1 Site Background Information

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion

records, well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling and water level measurement collection shall proceed from the least contaminated to the most contaminated well as indicated by previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

8.1.2 Groundwater Analysis Selection

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of quality assurance/quality control (QA/QC) samples to be collected (refer to the project-specific QAPP), as well as the type and volume of sample preservatives, the type and number of sample containers, the number of coolers required, and the quantity of ice or other chilling materials. The field sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Refer to the project-specific QAPP for the project analytical requirements.

8.2 Groundwater Sampling Procedures

Groundwater sampling procedures at a site shall include:

1) An evaluation of the well security and condition prior to sampling;
2) Decontamination of equipment;
3) Measurement of well depth to groundwater;
4) Assessment of the presence or absence of an immiscible phase;
5) Assessment of purge parameter stabilization;
6) Purging of static water within the well and well bore; and
7) Obtaining a groundwater sample.

Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events. In all cases, consult the State and local regulations for the site, which may require more stringent time separation between well development and sampling.

8.2.1 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information may be noted on a Groundwater Sample Collection Record (Attachment 1) or in the field logbook:

- Condition of the well's identification marker;
- Condition of the well lock and associated locking cap;
- Integrity of the well – well pad condition, protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing; and
- Condition of the general area surrounding the well.

8.2.2 Decontamination of Equipment

Where possible, dedicated supplies should be used at each well location to minimize the potential for cross-contamination and minimize the amount of investigation derived waste (IDW) fluids resulting from the decontamination process. If decontamination is necessary, establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with PE plastic sheeting and be located upwind of the well being sampled. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in SOP 3-06, Equipment Decontamination.

Decontaminate each piece of equipment prior to entering the well. Also, conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without on-site decontamination.

8.2.3 Measurement of Static Water Level Elevation

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. The best practice, if possible, is to measure all site wells (or wells within the monitoring well network) prior to sampling. If the well cap is not vented, remove the cap several minutes before measurement to allow water levels to equilibrate to atmospheric pressure.

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). If not already present, mark an easily identified reference point for water level measurements which will become the measuring point for all water level measurements. This location and elevation must be surveyed.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. A Teflon-free electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

At each location, measure water levels several times in quick succession to ensure that the well has equilibrated to atmospheric conditions prior to recording the measurement. As stated above, measure all site wells (or wells within the monitoring well network) prior to sampling whenever possible. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Prior to sampling, measure the water level in each well immediately prior to purging the well to ascertain that static conditions have been achieved prior to sampling.

8.2.4 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL before the well is purged for conventional sampling. These procedures may not be required for all wells. Consult the

project-specific QAPP to determine if assessing the presence of LNAPL and/or DNAPL is necessary.

1) Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a PID or an organic vapor analyzer, and record the measurements.

2) Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.

3) Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.

4) In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water table contour maps until they are corrected for depression by the product.

5) If the well contains an immiscible phase, it may be desirable to sample this phase separately. Section 8.2.6 presents immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the **TO Manager** and **Program Quality Manager** if this situation is encountered.

8.2.5 Purging Equipment and Use

General Requirements

The water present in a well prior to sampling may not be representative of in situ groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the IDW handling procedures in SOP 3-05, IDW Management. Purging shall be accomplished by methods as indicated in the project-specific QAPP or by those required by State requirements. For the purposes of this SOP, purging methods will be described by removing groundwater from the well using low-flow techniques.

According to the U.S. Environmental Protection Agency (EPA) (EPA, 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/minute. EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated corrosion or reaction products in the well. EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. In addition, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of increasing the turbidity of the sample due to mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The field sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells should not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated based on the results of either the well development task or historical sampling events. In general, place the intake of the purge pump in the middle of the saturated screened interval within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified. If the well is purged dry, allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis during well purging and analyze them in the field preferably using in-line devices (i.e., flow through cell) for temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation-reduction (redox) potential. Turbidity should be measured separately (outside of the flow-through cell) with a nephelometer or similar device.

Readings should be taken every 2 to 5 minutes during the purging process. These parameters are measured to demonstrate that the natural character of the formation waters has been restored.

Purging shall be considered complete per the requirements set forth in the project-specific QAPP, State requirements, or when three consecutive field parameter measurements of temperature, pH, specific conductivity, DO and ORP stabilize within approximately 10 percent and the turbidity is at or below 10 nephelometric turbidity units (NTU) or within ± 10% if above 10 NTU. This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process into a groundwater sampling log. Attachment 1 shows an example of a groundwater sampling log and the information typically included in the form. Whatever form is used, all blanks need to be completed on the field log during field sampling.

Groundwater removed during purging shall be stored according to the project-specific QAPP or per SOP 3-05, IDW Management.

Purging Equipment and Methods

Submersible Pump

A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling the groundwater for semivolatile and non-volatile constituents, but are generally not preferred for VOCs unless there are no other options (e.g., well over 200 feet deep). For wells over 200 feet deep, the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to overpumpage of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to limit the amount of water that will flow back down the drop pipe into the well. Place the pump in the middle of the saturated screened interval within the well and maintain it in that position during purging.

Bladder Pump

A stainless-steel bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Use of the bladder pump is most effective in low to moderate yield wells and are often the preferred method for low-flow sampling.

When sampling for VOCs and/or SVOCs and PFAS, polyethylene bladders and PFAS-free O-rings and pump accessories should be used.

Either compressed dry nitrogen or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use.

Centrifugal, Peristaltic, or Diaphragm Pump

A centrifugal, peristaltic, or diaphragm pump may be utilized to purge a well if the water level is within 20 feet of ground surface. New or dedicated HDPE tubing is inserted into the midpoint of the saturated screened interval of the well. Water should be purged at a rate that satisfies low-flow requirements (i.e., does not cause drawdown). Centrifugal, peristaltic, or diaphragm pump are generally discouraged for VOCs sampling; however, follow methods allowed per the project-specific QAPP or State requirements.

Air Lift Pump

Airlift pumps are not appropriate for purging or sampling.

Bailer

Avoid using a bailer to purge a well because it can result in overdevelopment of the well and create excessive purge rates. If a bailer must be used, the bailer should either be dedicated or disposable. An HDPE balier with polypropylene string mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well.

8.2.6 Monitoring Well Sampling Methodologies

Sampling Light, Non-Aqueous Phase Liquids (LNAPL)

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. Teflon bailers should always be used when sampling LNAPL. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

Groundwater Sampling Methodology

The well shall be sampled when groundwater within it is representative of aquifer conditions per the methods described in Section 8.2.5. Prior to sampling the flow-through cell shall be removed and

the samples collected directly from the purge tubing. Flow rates shall not be adjusted once aquifer conditions are met. Additionally, a period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling in order to monitor drawdown when using low-flow techniques and gauge well volumes removed and recharged when using non-low-flow techniques.

Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers, minimizing exposure to sunlight, and immediately placing the sample on ice once collected.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride (PVC) bailers, Tygon® tubing, silicon rubber bladders, neoprene impellers, polyethylene, and Viton® are not acceptable when sampling for organics and PFAS. If bailers are used, an inert cable/chain (e.g., polypropylene string or stainless steel wire or cable) shall be used to raise and lower the bailer. Dedicated equipment is highly recommended for all sampling programs.

Submersible Pumps

The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and HDPE, sample discharge lines composed of HDPE) and must have a controller mechanism allowing the required low-flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low-flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low-flow rate.

Bladder Pumps

A gas-operated stainless steel bladder pump with adjustable flow control and equipped with a polyethylene bladder and HDPE tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. If only inorganics are being sampled, polyvinyl bladders and tubing may be used. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge.

When using a compressor, take several precautions. If the compressor is being powered by a gasoline generator, position the generator downwind of the well. Ground fault circuit interrupters (GFCIs) should always be used when using electric powered equipment. Do not connect the compression hose from the compressor to the pump controller until after the engine has been started.

When all precautions are completed and the compressor has been started, connect the compression hose to the pump controller. Slowly adjust the control knobs to discharge water in the shortest amount of time while maintaining a near constant flow. This does not mean that the compressor must be set to discharge the water as hard as possible. The optimal setting is one that produces the largest volume of purge water per minute (not per purge cycle) while maintaining a near constant flow rate.

Prior to sampling, adjust the flow rate (purge rate) to yield 100 to 300 mL/minute. Avoid settings that produce pulsating streams of water instead of a steady stream if possible. Operate the pump at this low flow rate for several minutes to ensure that drawdown is not occurring. At no time shall the sample flow rate exceed the flow rate used while purging.

For those samples requiring filtration, it is recommended to use an in-line high capacity filter after all non-filtered samples have been collected.

Peristaltic Pumps:

A peristaltic pump is a type of positive displacement pump that moves water via the process of peristalsis. The pump uses a flexible hose fitted inside a circular pump casing. A rotor with cams compresses the flexible tube as the rotor turns, which forces the water to be pumped to move through the tube. In peristaltic pumps, no moving parts of the pump are in contact with the water being pumped. Displacement is determined by tube size, so delivery rate can only be changed during operation by varying pump speed. Peristaltic pumps are simple and quite inexpensive for the flow rates they provide.

There are several methods available for transferring the sample into the laboratory containers. The selected method may vary based on State requirements and should be documented in the project-specific QAPP. Samples typically can be collected directly from the discharge end of the HDPE tubing, after it has been disconnected from the flow through cell. For volatile analyses, the sampler should make sure that the pump is set such that a smooth laminar flow is achieved. In all cases, the project team should consult their local regulatory requirements and document the selected sample collection procedure in the project-specific QAPP.

Bailers

A single- or double-check valve HDPE or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical, regulatory, or logistical reasons.

Dedicated or disposable bailers should always be used in order to eliminate the need for decontamination and to limit the potential of cross-contamination. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

8.2.7 Sample Handling and Preservation

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable and require preservation. The U.S. EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods (SW-846)* (EPA 1997), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 provides guidance on the types of sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory or State requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field sampling personnel may add preservatives in the field.

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the project-specific QAPP. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)
2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Nitrate and ammonia
10. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. The analytical laboratory performing the analysis shall provide these vials. Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that has high concentrations of dissolved gasses. In these cases, the field sampling personnel shall document the occurrence in the field logbook and/or sampling worksheet at the time the sample was collected. Likewise, the analytical laboratory shall note in the laboratory analysis reports any headspace in the sample container(s) at the time of receipt by the laboratory.

Special Handling Considerations

In general, samples for organic analyses should not be filtered. However, high turbidity samples for PCB analysis may require filtering. Consult the project-specific QAPP for details on filtering requirements. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples should be handled in the same manner as VOC samples.

When collecting total and dissolved metals samples, the samples should be collected sequentially. The total metals sample is collected from the pump unfiltered. The dissolved metals sample is collected after filtering with a 0.45-micron membrane in-line filter. Allow at least 500 mL of effluent to flow through the filter prior to sampling to ensure that the filter is thoroughly wetted and seated in the filter capsule. If required by the project-specific QAPP, include a filter blank for each lot of filters used and always record the lot number of the filters.

Because there is some evidence that PFOS may sorb onto glass fiber filters, it is preferred not to filter samples for PFAS analysis in the field or laboratory. Field filtration is generally prohibited unless specifically requested by a client. If filtering is required by client's and regulatory agency's request, it is recommended that the following be considered and discussed with the client and regulatory agency:

- Evaluate if filtered results are meaningful, and, therefore, if filtering in the field or laboratory is required;

- Consider use of low flow sampling in the field to reduce the need for sample filtering;
- Consider use of a centrifuge in the laboratory to reduce the need for sample filtering; and
- If filtering is required, determine the nature of the filters used and do not use glass fiber filters.

Field Sampling Preservation

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. Guidance for the preservation of environmental samples can be found in the U.S. EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other U.S. EPA documents (EPA 1992, 1996).

Field Sampling Log

A groundwater sampling log provided as Attachment 1 shall document the following:

- Identification of well;
- Well depth;
- Static water level depth and measurement technique;
- Presence of immiscible layers and detection method;
- Well yield;
- Purge volume and pumping rate;
- Time that the well was purged;
- Sample identification numbers;
- Well evacuation procedure/equipment;
- Sample withdrawal procedure/equipment;
- Date and time of collection;
- Types of sample containers used;
- Preservative(s) used;
- Parameters requested for analysis;
- Field analysis data;
- Field observations on sampling event;
- Name of sampler; and
- Weather conditions.

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific QAPP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific QAPP will provide requirements for sample preservation and holding times,

container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

10.0 Data and records management

10.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:

- Sample Collection Records;
- Non-water repellent field logbook;
- Chain-of-custody forms; and
- Shipping labels.

10.2 Sample collection records (Attachment 1) will provide descriptive information for the purging process and the samples collected at each monitoring well.

10.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.

10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.

10.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

11.0 Attachments or References

Attachment 1 – Groundwater Sampling Collection Record

ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites.* ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. www.astm.org.

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater.* EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

EPA. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

EPA. 1997. *Test Methods for Evaluating Solid Waste, Physical/Chemical Method (SW-846)*. 3rd ed., Final Update IIIA. Office of Solid Waste. Online updates at: http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm.

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*.

SOP 3-05, *IDW Management.*

SOP 3-06, *Equipment Decontamination*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley, Environmental Scientist	Richard Purdy, Project Scientist	Rev 2 – Update & Review (June 2022)

Attachment 1
Groundwater Sample Collection Record

Well ID:

Groundwater Sample Collection Record

Client: _____ Date: _____ Time: Start _____ am/pm
Project No: _____ Finish _____ am/pm
Site Location: _____
Weather Conds: _____ Collector(s): _____

1. **WATER LEVEL DATA: (measured from Top of Casing)**
   a. Total Well Length _____ c. Length of Water Column _____ (a-b)
   b. Water Table Depth _____ d. Calculated Well Volume (see back) _____
   Casing Diameter/Material _____

2. **WELL PURGEABLE DATA**
   a. Purge Method: _____
   b. Acceptance Criteria defined (see SAP or Work Plan)
   - Minimum Required Purge Volume (@ _____ well volumes) _____
   - Maximum Allowable Turbidity _____ NTUs
   - Stabilization of parameters _____ %
   c. Field Testing Equipment used: Make Model Serial Number

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

(continued on back)

   d. Acceptance criteria pass/fail — Yes ☐ No ☐ N/A ☐
   Has required volume been removed
   Has required turbidity been reached
   Have parameters stabilized
   If no or N/A - Explain below.
   _____

3. **SAMPLE COLLECTION:** Method: _____

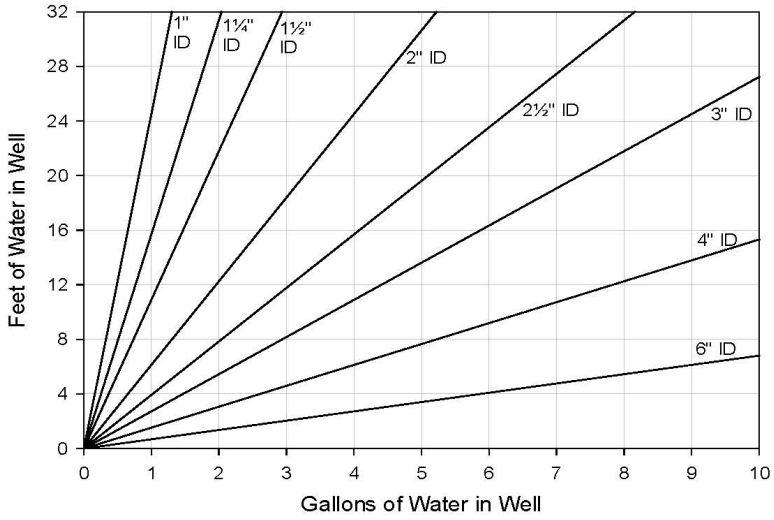
Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments _____

Signature _____ Date _____

Purge Volume Computation

Well ID:



Volume / Linear Ft. of Pipe		
ID (in)	**Gallon**	**Liter**
¼	0.0025	0.0097
3/8	0.0057	0.0217
½	0.0102	0.0386
¾	0.0229	0.0869
1	0.0408	0.1544
1¼	0.0637	0.2413
1½	0.0918	0.3475
2	0.1632	0.6178
2½	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

Signature _____ Date _____

AECOM

Soil and Rock Classification

Procedure 3-16

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) to thoroughly describe the physical characteristics of the sample and classify it according to the Unified Soil Classification System (USCS).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project-specific Quality Assurance Project Plan (QAPP).

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the Program Quality Manager. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling. All **field sampling personnel** responsible for sampling activities must review the project-specific Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP), paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations, and shall always be available on site.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety and Health Officer (SSHO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP/SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSHO.

2.4 The health and safety considerations for the work associated with soil classification include:

- At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.

- Stay clear of all moving equipment and be aware of pinch points on machinery. Avoid wearing loose fitting clothing.
- When using cutting tools, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and in case of extreme cold, wear insulating clothing.

3.0 Terms and Definitions

None.

4.0 Interference

None.

5.0 Training and Qualifications

5.1 The **Task Order (TO) Manager** is responsible for ensuring that the soil and rock classification procedures comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.3 The **Site Supervisor (SS)** is responsible for ensuring that all project **field personnel** follow these procedures.

5.4 Field personnel are responsible for the implementation of this procedure. Minimum qualifications for **field sampling personnel** require that one individual on the field team shall have a minimum of 6 months of experience with soil and rock classification.

5.5 The **project geologist** and/or **task manager** is responsible for directly supervising the soil and rock classification procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the **Program Quality Manager** and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials which may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- Personal protective equipment (PPE) and other safety equipment, as required by the APP/SSHP
- Field log book and pen with indelible ink
- Boring log
- Munsell Soil Color Chart
- Scoopula, spatula, and/or other small hand tools
- California Sampler
- Hand-held penetrometer

7.0 Calibration or Standardization

None.

8.0 Procedure

8.1 Soil Classification

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The USCS was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log, logbook, and/or electronic field data collection device. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand);
- Color, moisture, and odor;
- Range of particle sizes and maximum particle size;
- Approximate percentage of boulders, cobbles, gravel, sand, and fines;
- Plasticity characteristics of the fines;
- In-place conditions, such as consistency, density, and structure; and
- USCS classification symbol.

The USCS serves as ”shorthand“ for classifying soil into 15 basic groups:

GW[1] Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines);

GP[1] Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines);

GM[1] Silty gravel (>50 percent gravel, >15 percent silt);

GC[1] Clayey gravel (>50 percent gravel, >15 percent clay);

SW[1] Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines);

SP[1] Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines);

SM[1] Silty sand (>50 percent sand, >15 percent silt);

SC[1] Clayey sand (>50 percent sand, >15 percent clay);

[1] If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

ML[2] Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity);

CL[2] Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity);

MH[2] Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity);

CH[2] Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity);

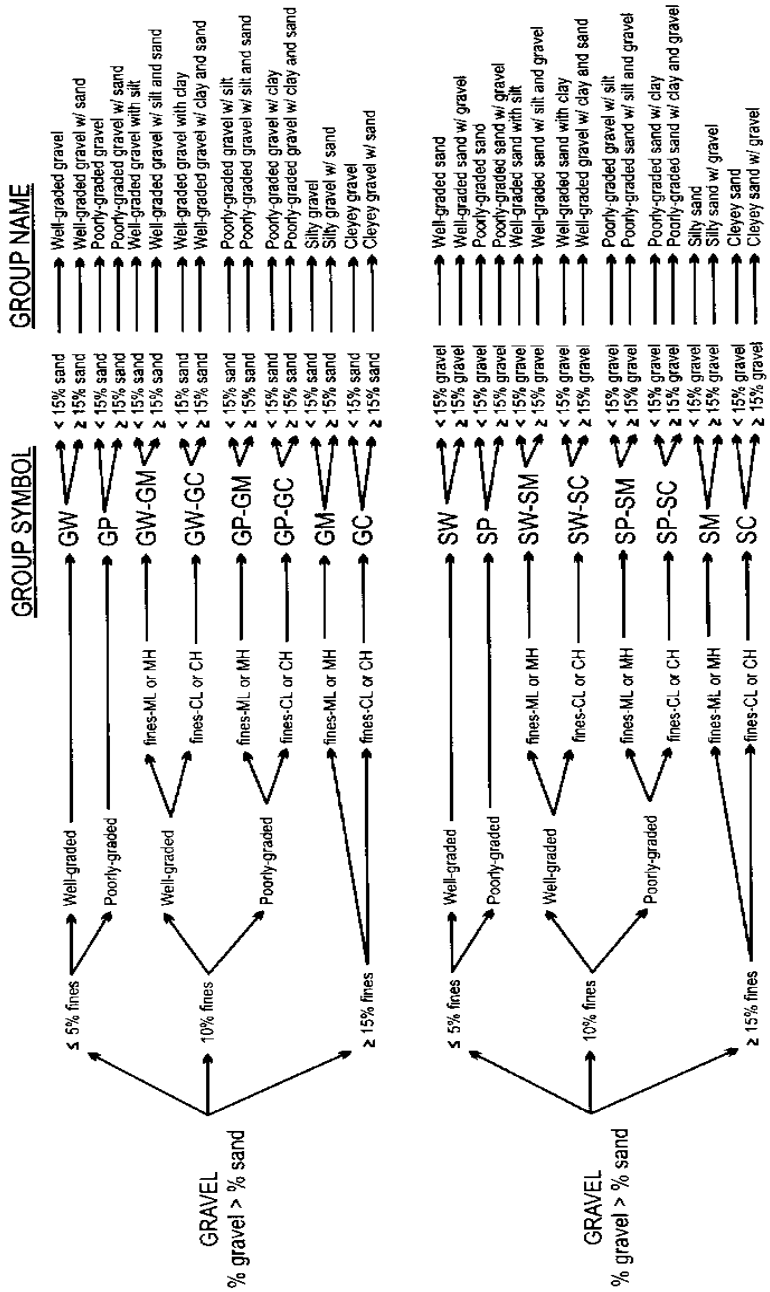
OL Organic low plasticity silt or organic silty clay;

OH Organic high plasticity clay or silt; and

PT Peat and other highly organic soil.

[2] If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words “with sand” or “with gravel” (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words “sandy” or “gravely” (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use “sandy.”

Figure 8-1 defines the terminology of the USCS. Flow charts presented in Figure 8-2 and



indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.

8.1.1 Estimation of Particle Size Distribution

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and

frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.
3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term “trace”.
7. The percentages of gravel, sand, and fines must add up to 100 percent. “Trace” is not included in the 100 percent total.

8.1.2 Soil Dilatancy, Toughness, and Plasticity

8.1.2.1 **Dilatancy**

To evaluate dilatancy, follow these procedures:

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 8-1. The reaction is the speed with which water appears while shaking and disappears while squeezing.

Table 8-1: Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

8.1.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 8-2.

Table 8-2: Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

DEFINITION OF TERMS					
MAJOR DIVISIONS			SYMBOLS		TYPICAL DESCRIPTIONS
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)		GW	Well graded gravels, gravel-sand mixtures, little or no fines
				GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS With Fines		GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines
				GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)		SW	Well graded sands, gravelly sands, little or no fines
				SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS With Fines		SM	Silty sands, sand-silt mixtures, non-plastic fines
				SC	Clayey sands, sand-clay mixtures, plastic fines
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%			ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines
				CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays
				OL	Organic silts and organic silty clays of low plasticity
	SILTS AND CLAYS Liquid Limit is Greater Than 50%			MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt
				CH	inorganic clays of high plasticity, fat clays
				OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS				PT	Peat and other highly organic soils

GRAIN SIZES								
SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS	
	FINE	MEDIUM	COARSE	FINE	COARSE			
200	40	10	4	3/4"	3"	12"		
U.S. STANDARD SERIES SIEVE				CLEAR SQUARE SIEVE OPENINGS				

Figure 8-1: Unclassified Soil Classification System (USCS)

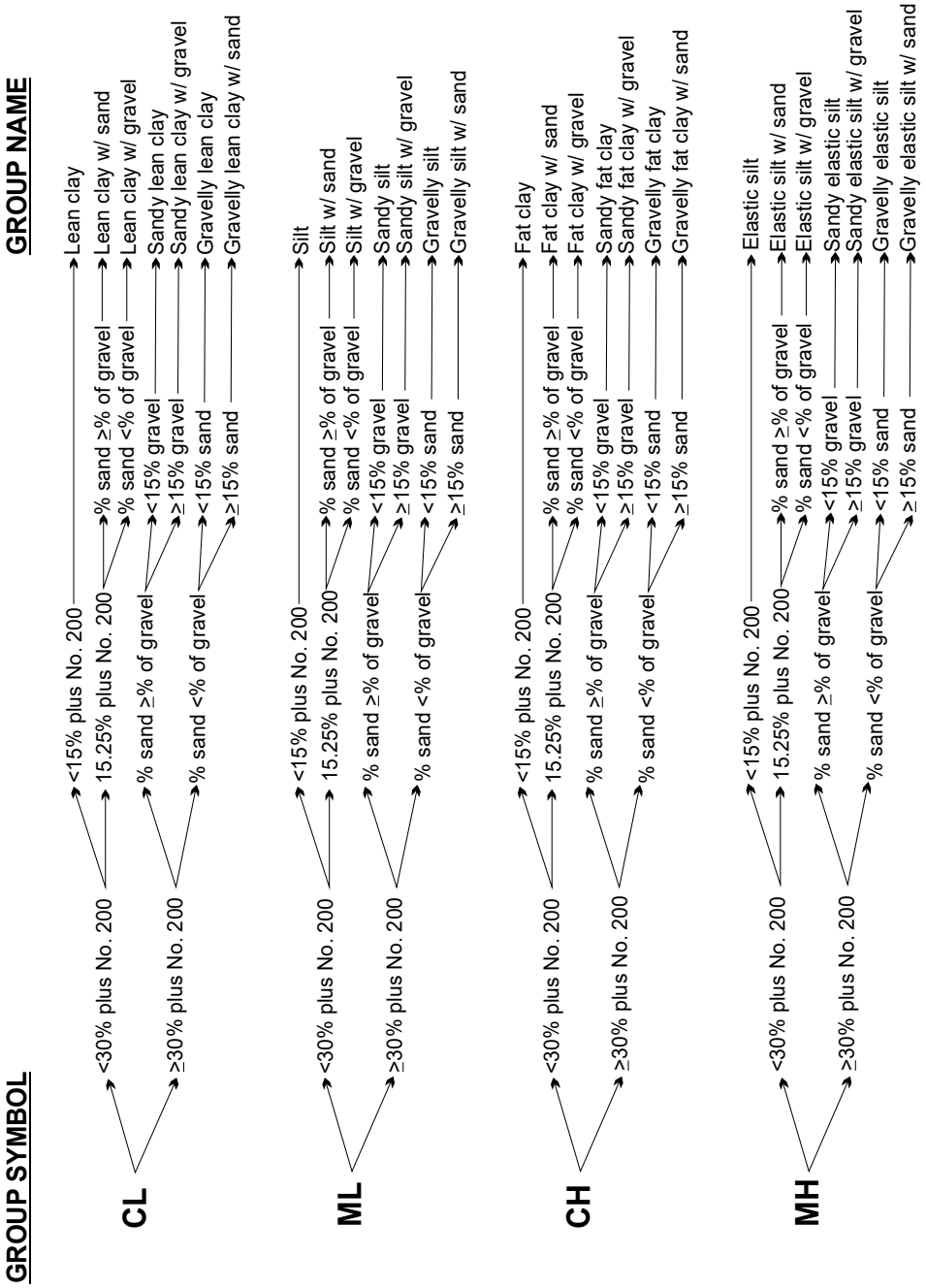


Figure 8-2: Flow Chart for Fine Grain Soil Classification

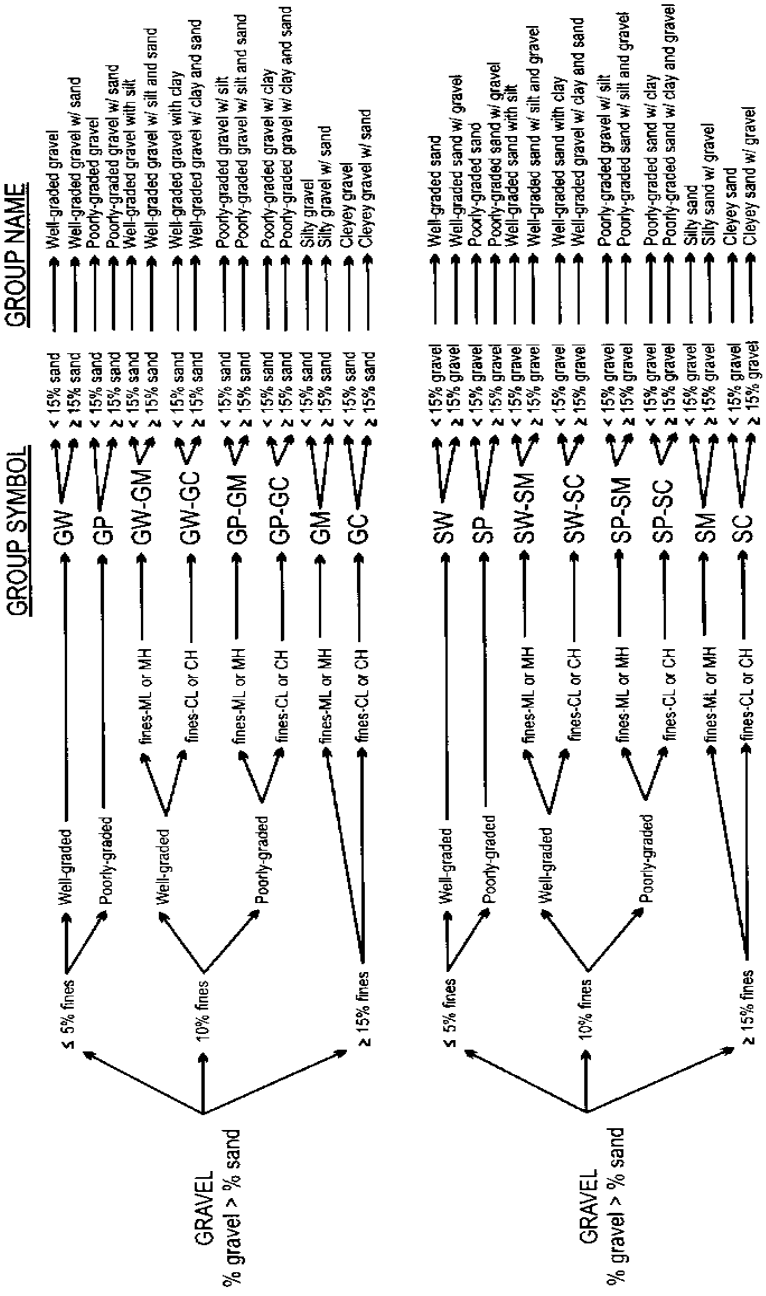


Figure 8-3: Flow Chart for Soil with Gravel

8.1.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 8-3 presents the criteria for describing plasticity in the field using the rolled thread method.

Table 8-3: Criteria for Describing Plasticity

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

8.1.3 Angularity

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides but have well-rounded corners and edges.
- **Subangular** particles are similar to angular but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

8.1.4 Color, Moisture, and Odor

The natural moisture content of soil is very important. Table 8-4 shows the terms for describing the moisture condition and the criteria for each.

Table 8-4: Soil Moisture Content Qualifiers

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table

Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like “5YR 5/6, yellowish-red.” Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

8.1.5 In-Place Conditions

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

8.1.5.1 Density/Consistency

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term “density” is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term “consistency” is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs. (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the “standard penetration resistance,” or the “N-value.” The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless-steel liners and penetrating a cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed “refusal” and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

  If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 8-5 and Table 8-6 present representative descriptions of soil density/consistency vs. N-values.

Table 8-5: Measuring Soil Density with a California Sampler – Relative Density (Sands, Gravels)

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.43 factor
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

Table 8-6: Measuring Soil Density with a California Sampler – Fine Grained Cohesive Soil

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or Shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 8-7):

Table 8-7: Measuring Soil Consistency with a Hand-Held Penetrometer

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8-8.

Table 8-8: Measuring Soil Consistency Using Thumb Pressure

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

8.1.5.2 Cementation

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

- Quartz – siliceous;
- Chert – chert-cemented or chalcedonic;
- Opal – opaline;
- Carbonate – calcitic, dolomitic, sideritic (if in doubt, calcareous should be used);
- Iron oxides – hematitic, limonitic (if in doubt, ferruginous should be used);
- Clay minerals – if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as “kaolin-cemented,” “chlorite-cemented,” etc.; and
- Miscellaneous minerals – pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

- Weak – friable; crumbles or breaks with handling or slight finger pressure;
- Moderate – friable; crumbles or breaks with considerable finger pressure; and
- Strong – not friable; will not crumble or break with finger pressure.

8.1.5.3 Structure

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular – spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified – alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated – alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky – cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed – inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar – particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy – particles are arranged about a horizontal plane

8.1.5.4 Other Features

- Mottled – soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured – breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)
- Slickensided – fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

8.1.6 Development of Soil Description

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

8.1.6.1 Coarse-grained Soil

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is “sand-sized.” It is classified as a gravel if over 50 percent of the coarse fraction is composed of “gravel-sized” particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

8.1.6.2 Fine-grained Soil

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.

Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

8.1.6.3 Organic Soil

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

8.2 Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

8.3 Rock Classification

The purpose of rock classification is to thoroughly describe the physical and mineralogical characteristics of a specimen and to classify it according to an established system. The generalized rock classification system described below was developed because, unlike the USCS for soils, there is no universally accepted rock classification system. In some instances, a more detailed and thorough rock classification system may be appropriate. Any modifications to this classification system, or the use of an alternate classification system should be considered during preparation of the site work plan. Both the TO Manager and the QA Manager or Technical Director must approve any modifications to this classification system, or the use of another classification system.

Describing rock specimens on a common basis is essential so that rocks described by different site geologists are comparable. Site geologists describing rock specimens as a part of investigative activities must use the classification system described herein, or if necessary, another more detailed classification system. Use of a common classification system provides the most useful geologic database for all present and future subsurface investigations and remedial activities.

In order to provide a more consistent rock classification between geologists, a rock classification template has been designated as shown in Figure 8-4. The template includes classification of rocks by origin and mineralogical composition. When classifying rocks, all site geologists shall use this template.

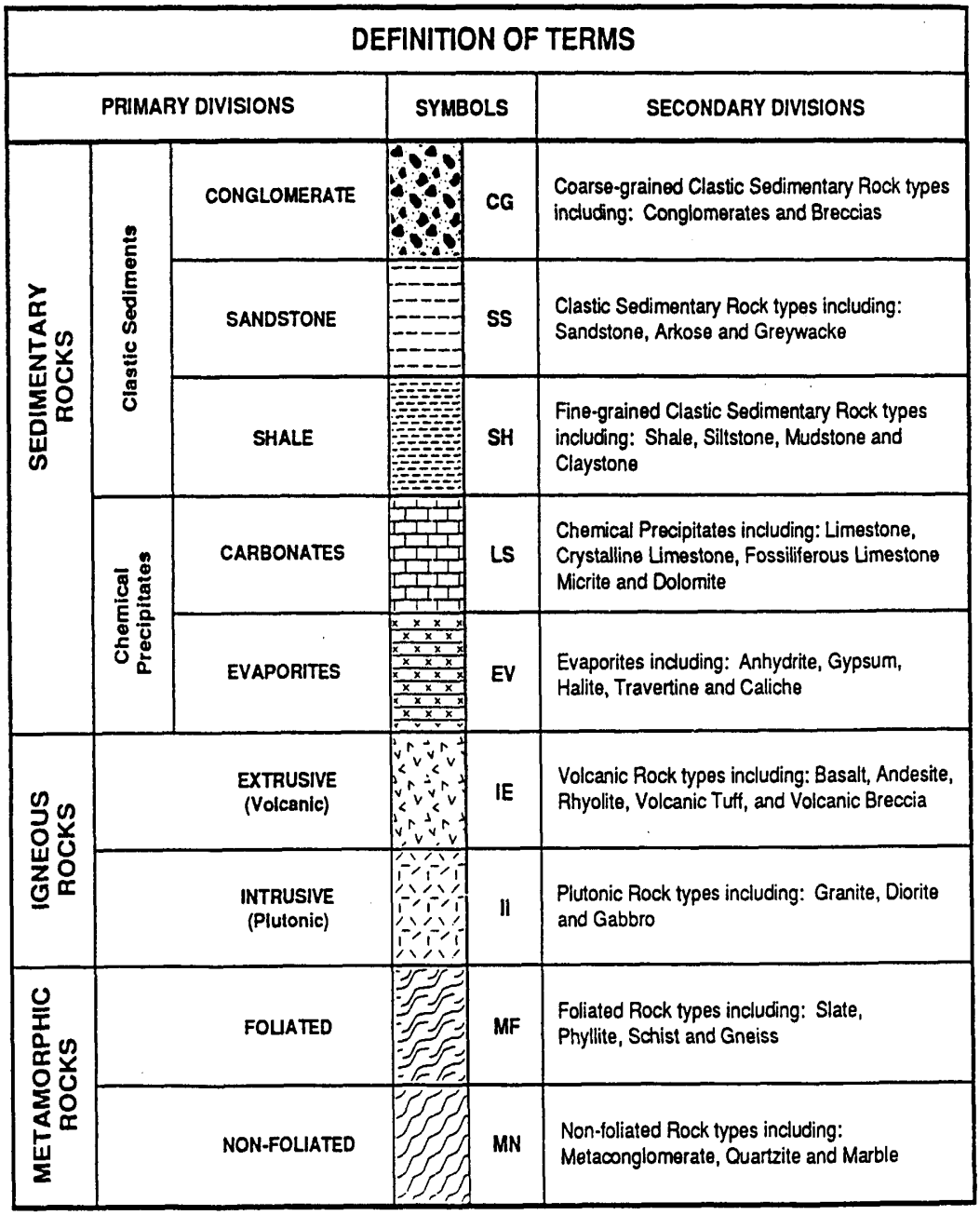
The site geologist shall describe the rock specimen and record the description in a boring log or logbook. The items essential for classification include (i.e., metamorphic foliated):

- Classification Name (i.e., schist);
- Color;
- Mineralogical composition and percent;
- Texture/Grain size (i.e., fine-grained, pegmatitic, aphlitic, glassy);
- Structure (i.e., foliated, fractured, lenticular);
- Rock Quality Designation (sum of all core pieces greater than two times the diameter of the core divided by the total length of the core run, expressed as a percentage); and
- Classification symbol (i.e., MF).

Example: Metamorphic foliated schist: Olive gray, 5Y, 3/2, Garnet 25 percent, Quartz 45 percent, Chlorite 15 percent, Tourmaline 15 percent, Fine-grained with Pegmatite garnet, highly foliated, slightly wavy, MF.

9.0 Quality Control and Assurance

None



DEFINITION OF TERMS					
PRIMARY DIVISIONS				SYMBOLS	SECONDARY DIVISIONS
SEDIMENTARY ROCKS	Clastic Sediments	CONGLOMERATE		CG	Coarse-grained Clastic Sedimentary Rock types including: Conglomerates and Breccias
		SANDSTONE		SS	Clastic Sedimentary Rock types including: Sandstone, Arkose and Greywacke
		SHALE		SH	Fine-grained Clastic Sedimentary Rock types including: Shale, Siltstone, Mudstone and Claystone
	Chemical Precipitates	CARBONATES		LS	Chemical Precipitates including: Limestone, Crystalline Limestone, Fossiliferous Limestone Micrite and Dolomite
		EVAPORITES		EV	Evaporites including: Anhydrite, Gypsum, Halite, Travertine and Caliche
IGNEOUS ROCKS	EXTRUSIVE (Volcanic)			IE	Volcanic Rock types including: Basalt, Andesite, Rhyolite, Volcanic Tuff, and Volcanic Breccia
	INTRUSIVE (Plutonic)			II	Plutonic Rock types including: Granite, Diorite and Gabbro
METAMORPHIC ROCKS	FOLIATED			MF	Foliated Rock types including: Slate, Phyllite, Schist and Gneiss
	NON-FOLIATED			MN	Non-foliated Rock types including: Metaconglomerate, Quartzite and Marble

Figure 8-4: Rock Classification System

10.0 Data and Records Management

10.1 Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be sent to the **TO Manager** for the project files.

10.2 Field notes will be kept during coring activities in accordance with SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody. The information pertinent to soil classification activities includes chronology of events, sample locations (x,y,z), time/date, sampler name, methods (including type of core liner/barrel, if applicable), sampler penetration and acceptability, sample observations, and the times and type of equipment decontamination. Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

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Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley, Environmental Scientist	Richard Purdy, Project Scientist	Rev 2 – Update & Review (June 2022)

Operation and Calibration of a Photoionization Detector

Procedure 3-20

1.0 Purpose and Scope

1.1 Purpose and Applicability

1.1.1 This standard operating procedure (SOP) describes the procedures that will be followed by field staff for operation and calibration of a photoionization detector (PID). The PID is primarily used by AECOM personnel for safety and survey monitoring of ambient air, determining the presence of volatiles in soil and water, and detecting leakage of volatiles.

1.1.2 PIDs routinely used by field personnel include the Photovac Microtip, Thermoelectron 580EZ, MiniRAE 2000, and MiniRae 3000. Personnel responsible for using the PID should first read and thoroughly familiarize themselves with the instrument instruction manual.

1.2 Principle of Operation

1.2.1 The PID is a non-specific vapor/gas detector. The unit generally consists of a hand-held probe that houses a PID, consisting of an ultraviolet (UV) lamp, two electrodes, and a small fan which pulls ambient air into the probe inlet tube. The probe is connected to a readout/control box that consists of electronic control circuits, a readout display, and the system battery. Units are available with UV lamps having an energy from 9.5 electron volts (eV) to 11.7 eV.

1.2.2 The PID analyzer measures the concentration of trace gas present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule (in electron volts (eV) is less than the energy of the photon. The source of photons is an ultraviolet lamp in the probe unit. Lamps are available with energies ranging from 9.5 eV to 11.7 eV. All organic and inorganic vapor/gas compounds having ionization potentials lower than the energy output of the UV lamp are ionized and the resulting potentiometric change is seen as a positive reading on the unit. The reading is proportional to the concentration of organics and/or inorganics in the vapor.

1.2.3 Sample gases enter the probe through the inlet tube and enter the ion chamber where they are exposed to the photons emanating from the UV lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive-biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This current is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

1.2.4 In service, the analyzer is first calibrated with a gas of known composition equal to, close to, or representative of that to be measured. Gases with ionization potentials near to or less than the energy of the lamp will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials greater than the energy of the lamp will not be detected. The ionization potentials of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to 15.6 eV and are not ionized by any of the lamps available. Gases with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

1.3 Specifications

1.3.1 Refer to the manufacturer's instructions for the technical specifications of the instrument being used. The operating concentration range is typically 0.1 to 2,000 ppm isobutylene equivalent.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan (SSHP). Work will also be conducted according to the Task Order (TO) Quality Assurance Project Plan (QAPP) and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Only PIDs stamped Division I Class I may be used in explosive atmospheres. Refer to the project APP/SSHP for instructions pertaining to instrument use in explosive atmospheres.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Regardless of which gas is used for calibration, the instrument will respond to all analytes present in the sample that can be detected by the type of lamp used in the PID.

4.2 Moisture will generate a positive interference in the concentration measured for a PID and is characterized by a slow increase in the reading as the measurement is made. Care must be taken to minimize uptake of moisture to the extent possible. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.3 Uptake of soil into the PID must be avoided as it will compromise instrument performance by blocking the probe, causing a positive interference, or dirtying the PID lamp. Refer to the manufacturers' instructions for care, cleaning, and maintenance.

4.4 The user should listen to the pitch of the sampling pump. Any changes in pitch may indicate a blockage and corrective action should be initiated.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that the operation and calibration activities comply with this procedure. The **TO Manager** is responsible for ensuring that all personnel involved in the operation and calibration shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all operation and calibration activities are conducted according to this procedure.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

- Calibration Gas: Compressed gas cylinder of isobutylene in air or similar stable gas mixture of known concentration. The selected gas should have an ionization potential similar to that of the vapors to be monitored, if known. The concentration should be at 50-75% of the range in which the instrument is to be calibrated
- Regulator for calibration gas cylinder
- Approximately 6 inches of Teflon® tubing
- Tedlar bag (optional)
- Commercially-supplied zero grade air (optional)
- "Magic Marker" or “Sharpie” or other waterproof marker
- Battery charger
- Moisture traps
- Spare lamps
- Manufacturer’s instructions
- Field data sheets or logbook/pen

7.0 Procedure

7.1 Preliminary Steps

7.1.1 Preliminary steps (battery charging, check-out, calibration, maintenance) should be conducted in a controlled or non-hazardous environment.

7.2 Calibration

7.2.1 The PID must be calibrated in order to display concentrations in units equivalent to ppm. First a supply of zero air (ambient air or from a supplied source), containing no ionizable gases or vapors is used to set the zero point. A span gas, containing a known concentration of a photoionizable gas or vapor, is then used to set the sensitivity.

7.2.2 Calibrate the instrument according to the manufacturer’s instructions. Record the instrument model and identification number, the initial and adjusted meter readings, the calibration gas composition and concentration, and the date and the time in the field records.

7.2.3 If the calibration cannot be achieved or if the span setting resulting from calibration is 0.0, then the lamp must be cleaned (Section 7.4).

7.3 Operation

7.3.1 Turn on the unit and allow it to warm up (minimum of 5 minutes). Check to see if the intake fan is functioning; if so, the probe will vibrate slightly and a distinct sound will be audible when holding the probe casing next to the ear. Also, verify on the readout display that the UV lamp is lit.

7.3.2 Calibrate the instrument as described in Section 7.2, following the manufacturer’s instructions. Record the calibration information in the field records.

7.3.3 The instrument is now operational. Readings should be recorded in the field records.

7.3.4 When the PID is not being used or between monitoring intervals, the unit may be switched off to conserve battery power and UV lamp life; however, a “bump” test should be performed each time the unit is turned on and prior to taking additional measurements. To perform a

bump test, connect the outlet tubing from a Tedlar bag containing a small amount of span gas to the inlet tubing on the unit and record the reading. If the reading is not within the tolerance specified in the project plan, the unit must be recalibrated.

7.3.5 At the end of each day, recheck the calibration. The check will follow the same procedures as the initial calibration (Section 7.2) except that no adjustment will be made to the instrument. Record the information in the field records.

7.3.6 Recharge the battery after each use (Section 7.4).

7.3.7 When transporting, ensure that the instrument is packed in its stored condition in order to prevent damage.

7.4 Routine Maintenance

7.4.1 Routine maintenance associated with the use of the PID includes charging the battery, cleaning the lamp window, replacing the detector UV lamp, replacing the inlet filter, and replacing the sample pump. Refer to the manufacturer's instructions for procedures and frequency.

7.4.2 All routine maintenance should be performed in a non-hazardous environment.

7.5 Troubleshooting Tips

7.5.1 One convenient method for periodically confirming instrument response is to hold the sensor probe next to the tip of a magic marker. A significant reading should readily be observed.

7.5.2 Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings.

7.5.3 A fogged or dirty lamp, due to operation in a humid or dusty environment, may cause erratic or fluctuating readings. The PID should never be operated without the moisture trap in place.

7.5.4 Moving the instrument from a cool or air-conditioned area to a warmer area may cause moisture to condense on the UV lamp and produce unstable readings.

7.5.5 A zero reading on the meter should not necessarily be interpreted as an absence of air contaminants. The detection capabilities of the PID are limited to those compounds that will be ionized by the particular probe used.

7.5.6 Many volatile compounds have a low odor threshold. A lack of meter response in the presence of odors does not necessarily indicate instrument failure.

7.5.7 When high vapor concentrations enter the ionization chamber in the PID the unit can become saturated or "flooded". Remove the unit to a fresh air environment to allow the vapors to be completely ionized and purged from the unit.

8.0 Quality Control and Assurance

8.1 The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific QAPP.

8.2 Calibration of the PID will be conducted at the frequency specified in the project plan. In the absence of project-specific guidance, calibration will be performed at the beginning of each day of sampling and will be checked at the end of the sampling day or whenever instrument operation is suspect. The PID will sample a calibration gas of known concentration. The instrument must agree with the calibration gas within ±10%. If the instrument responds outside this tolerance, it must be recalibrated.

8.3 Checks of the instrument response (Section 7.5) should be conducted periodically and documented in the field records.

9.0 Records, Data Analysis, Calculations

Safety and survey monitoring with the PID will be documented in a bound field logbook, or on standardized forms, and retained in the project files. The following information is to be recorded:

- Project name and number;
- Instrument manufacturer, model, and identification number;
- Operator's signature;
- Date and time of operation;
- Calibration gas used;
- Calibration check at beginning and end of day (meter readings before adjustment);
- Span setting after calibration adjustment;
- Meter readings (monitoring data obtained);
- Instances of erratic or questionable meter readings and corrective actions taken; and
- Instrument checks and response verifications – e.g., battery check, magic marker response (Section 7.5) or similar test.

10.0 Attachments or References

United States Environmental Protection Agency. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM). USEPA, Region 4, SESD, Enforcement and Investigations Branch, Athens, GA. November 2001.

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley, Environmental Scientist	Richard Purdy, Project Scientist	Rev 2 – Update & Review (June 2022)

AECOM

Surface and Subsurface Soil Sampling Procedures

Procedure 3-21

1.0 Purpose and Scope

1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, sonic drilling, and split-spoon equipment.

1.2 For project specific information (e.g., sampling depths, equipment to be used, and frequency of sampling), refer to the Quality Assurance Project Plan (QAPP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by United States Environmental Protection Agency (USEPA) standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push, sonic drilling, and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials, such as medium to fine sands, silts, and silty clay soils. Sonic drilling sampling works well in all types of soil and bedrock. Split-spoon sampling works well in all types of soil but is somewhat slower than direct-push and sonic drilling methods. With the exception of volatile organic compounds (VOCs) samples, the soil sample interval is composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for VOC analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are best collected with direct push or by Shelby Tube (not covered in this SOP). They are collected, sealed, and sent directly to the laboratory for analysis without homogenizing.

2.0 Safety

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Accident Prevention Plan (APP)/Site Safety and Health Plan. Work will also be conducted according to the Task Order (TO) QAPP and/or direction from the **Site Safety and Health Officer (SSHO)**.

2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

3.0 Terms and Definitions

None.

4.0 Interferences

4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.

4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.

4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.

4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

5.0 Training and Qualifications

5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 Responsibilities

5.2.1 The **TO Manager** is responsible for ensuring that soil sampling activities comply with this procedure. The TO Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Site Supervisor (SS)** is responsible for ensuring that all soil sampling activities are conducted according to this procedure.

5.2.4 All **Field Personnel** are responsible for the implementation of this procedure.

6.0 Equipment and Supplies

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the QAPP. The following details equipment typically needed for soil sampling, based on the various methods. See the QAPP for specific detail of equipment and supply needs.

6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:

- PID/FID;
- Calibration gas; and
- Tedlar® gas bags (for calibration).

6.2 If field screening methods include jar headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:

- Clean soil (“drillers jars”) jars;

- HDPE sample jars for PFAS/PFOS samples; and
- Aluminum foil for non-PFAS/PFOS sampling.

6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:

- Alconox® or Liquinox®;
- Isopropyl Alcohol;
- Deionized Ultra-Filtered (DIUF) Water (confirmed PFAS-free);
- Plastic buckets or washbasins;
- Brushes; and
- Polyethylene sheeting.

6.4 The following general equipment is needed for all soil sampling, regardless of method:

- Stainless steel bowls;
- Stainless steel trowels;
- Appropriate sample containers for laboratory analysis;
- Personal Protective Equipment (PPE);
- Non-water-repellent logbook;
- Cooler and ice for preservation; and
- Stakes and flagging to document sampling location.

6.5 The following additional equipment is needed for volatile organic sampling:

- Electronic pan scale and weights for calibration; and
- Syringes or other discrete soil core samplers.

6.6 The following additional equipment may be needed for surface and test pit soil sampling:

- Hand Auger.

6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:

- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

7.0 Procedure

7.1 General Soil Sampling Procedure for All Soil Sampling Methods

7.1.1 Record the weather conditions and other relevant on-site conditions.

7.1.2 Select the soil sampling location, clear vegetation, if necessary, and record the sampling location identification number and pertinent location details.

7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e., tables with polyethylene sheeting).

7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, sonic drilling, direct push, or test pit sample collection (7.2, 7.3, 7.4, 7.5, and 7.6, respectively).

7.1.6 Collect soil samples according to procedures listed in Section 7.7 depending on project specific analyses.

7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.

7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.

7.1.9 Dispose of all excess excavated soil in accordance with the QAPP.

7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.

7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

7.2 Surface Sampling

7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:

- Visual observations (soil staining, fill materials);
- Other relevant soil characteristics;
- Site features;
- Screening results;
- Predetermined sampling approach (i.e., grid or random); and
- Sampling objectives as provided in the QAPP.

7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the QAPP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the QAPP.

1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
2. Remove soil from the exposed sampling area with a stainless-steel trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless-steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the APP/SSHP.
3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.7.1 for VOC sampling.

4. Collect sufficient soil to fill all remaining sample jars into a stainless-steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:

   a) Remove all rocks and non-soil objects using a stainless-steel spoon or scoop.

   b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.

   c) Use the stainless-steel spoon/scoop to mix the quarter samples that are opposite.

   d) After mixing the opposite quarters, reform the cone shaped mound.

   e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

7.3 Split-Spoon Sampling

7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the QAPP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.

7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.

7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the QAPP, VOC and headspace samples should be collected (see Section 7.7.1) prior to logging the sample.

7.3.4 If headspace screening for VOCs is required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the QAPP.

7.3.6 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.3.7 The QAPP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with polyethylene sheeting. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.4 Sonic Drilling Sampling

7.4.1 At each boring location, the frequency and depth of sonic drilling samples will be determined from the QAPP.

7.4.2 Sonic drilling methods, also known as vibratory drilling, use an eccentrically oscillating drill head to produce high-frequency vibratory energy that is then transmitted down a drill string to a core barrel to quickly advance through the subsurface. Sonic drilling utilizes a double-cased system using an inner core barrel and a larger override casing. This ensures that the borehole is continuously cased to the total depth, minimizing the potential for borehole collapse and providing the means to alter casing diameters to telescope through semi-confining units to prevent downhole cross contamination.

7.4.3 Upon retrieval of the core barrel, place the tubular plastic sleeve (confirmed PFAS-free) with sealed bottom over the bottom of the core barrel. The core barrel will then be vibrated, causing the soil sample to be extruded into the sleeve. Place the sleeve on the work surface (i.e. PFAS-free plastic covered table or ground). Open the sleeve and measure the recovery of soil.

7.4.4 If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the soil core and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the QAPP, VOC and headspace samples should be collected (see Section 7.7.1) prior to logging the sample.

7.4.5 If headspace screening for VOCs is required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.4.6 Soils collected using sonic drilling will be logged by the field representative using the procedure required in the QAPP.

7.4.7 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.4.8 The QAPP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from each interval will be set aside. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.

7.4.9 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.5 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the QAPP. Typically, samples with direct-push equipment are collected in 4-foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the QAPP.

2. Cut open the acetate liner. If required in the QAPP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID

inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the QAPP should be collected (see Section 7.7.1) prior to logging the sample.

3. If required in the QAPP, collect a soil sample (as defined in the QAPP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

4. Soils collected using the direct-push sampler will be logged by the by the field representative using the procedure required in the QAPP.

5. Collect the remainder of the sample into a stainless-steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the QAPP (e.g., completed as a monitoring well, backfilled with bentonite, etc.).

7.6 Test Pit Sampling

7.6.1 Excavate the test pit to the desired depth.

7.6.2 Using the excavator bucket, collect soil samples as specified in the QAPP. Collect a sample and perform screening analyses as required by the QAPP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.6.3 Collect the sample from center of the bucket to avoid potential contamination from the bucket.

7.6.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.6.5 Collect the remainder of the sample volume required into a stainless-steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.6.6 Dispose of all excavated soil according to the QAPP.

7.7 Sample Collection Methods

7.7.1 Volatile Organics Sampling

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgeable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:

   a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.

   b) Tare the sampler by placing it on the scale and zeroing the scale.

c) Draw back the plunger to the 5-gram mark or 5mL (5cc) mark on cut-off syringe and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.

d) Weigh the filled sampler and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.

2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.

3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.

4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.

5. Fill the other low level 40 mL VOA vial in this manner.

6. Repeat the process for the high-level VOA vials, only for the high-level VOA vial three 5-gram aliquots (i.e., 15 grams total) should be extruded into the high-level VOA vial.

   NOTE: Depending on the laboratory, some high-level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high-level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).

7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.

8. Fill the 4-oz glass jar with soil from the same area for percent moisture determination.

7.7.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least ¾ full for all other analyses. Homogenize the soil in a decontaminated stainless-steel bowl, removing rocks, sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

8.0 Quality Control and Assurance

8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the QAPP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the QAPP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.

8.2 Quality control requirements are dependent on project-specific sampling objectives. The QAPP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.

9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

10.0 Attachments or References

SOP 3-06, *Equipment Decontamination*

SOP 3-19, *Headspace Screening for Total VOCs*

SOP 3-20, *Operation and Calibration of a Photoionization Detector*

Author	***Reviewer***	***Revisions (Technical or Editorial)***
Robert Shoemaker, PMP Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)
Ken O'Donnell, PG Geologist	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Robert Shoemaker, PMP Senior Scientist	Josh Millard, PG, CPG	Rev 2 – Addition of Sonic Drilling Methods (January 2020)
Rose Kelley, Environmental Scientist	Richard Purdy, Project Scientist	Rev 3 – Update & Review (June 2022)

Water Quality Parameter Testing

Procedure 3-24

1.0 Purpose and Scope

1.1 The purpose of this document is to define the standard operating procedure (SOP) for use water quality parameter testing for groundwater or surface water sampling. This SOP describes the equipment, field procedures, materials, and documentation procedures necessary to complete this task. Specific information regarding coring locations can be found in the associated Quality Assurance Project Plan (QAPP).

1.2 This procedure is the Program-approved professional guidance for work performed by AECOM under the client contract.

1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review. If there are procedures whether it be from AECOM, state and/or federal that are not addressed in this SOP and are applicable to surface water sampling then those procedures may be added as an appendix to the project specific QAPP.

1.4 It is fully expected that the procedures outlined in this SOP will be followed. Procedural modifications may be warranted depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by the **Task Order (TO) Manager** or **Program Quality Manager**. Deviations to this SOP will be documented in the field records.

2.0 Safety

2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first surface water sampling location. All **field sampling personnel** responsible for sampling activities must review the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP), paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor and liquid phase through the use of respirators and disposable clothing.

2.2 In addition, observe standard health and safety practices according to the project-specific APP/SSHP. Suggested minimum protection during well sampling activities includes protective eyewear, powder-free nitrile gloves, rubberized steel-toed boots, and an American National Standards Institute-standard hard hat. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on evaluation for PFAS and on the contaminant concentrations.

2.3 Daily safety briefs will be conducted at the start of each working day before any work commences. These daily briefs will be facilitated by the **Site Safety Officer (SSO)** or designee to discuss the day's events and any potential health risk areas covering every aspect of the work to be completed. Weather conditions are often part of these discussions. As detailed in the APP/SSHP, everyone on the field team has the authority to stop work if an unsafe condition is perceived until the conditions are fully remedied to the satisfaction of the SSO.

2.4 The health and safety considerations for the work associated with surface water sampling include:

- Proper selection of personal protective equipment for work around water bodies (e.g., personal flotation devices [PFDs]), as specified in the project-specific APP/SSHP.

- Appropriate health and safety protocols for working in a boat (if applicable), as specified in the project-specific APP/SSHP.
- Proper lifting techniques when retrieving surface water samplers, large muscles of the legs should be used, not the back.
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
- To avoid slip/trip/fall hazards as a result of working on wet surfaces, wear work boots/work boot covers with textured soles.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended), and in cases of extreme cold, wear fitted insulated clothing

3.0 Terms and Definitions

3.1 **Barometric Pressure (BP)**: The density of the atmosphere, which varies according to altitude and weather conditions.

3.2 **Conductivity/Specific Conductance**: A measure of the ability of water to pass electrical current, which increases with the amount of dissolved ionic substances (i.e., salts). Conductivity is inversely related to the resistance of a solution and is measured in units of mhos per centimeter (mhos/cm) (inverse ohms/cm, Siemens/cm). The conductivity of water increases with increasing temperature. Specific Conductance is corrected for 25 degrees Celsius (°C); for this reason, it is best to record Specific Conductance. If Conductivity is recorded, the temperature of the sample MUST recorded.

3.3 **Dissolved Oxygen (DO):** The amount of oxygen present in water and available for respiration. DO is typically measured in milligrams per liter (mg/L). Oxygen is less soluble in warm and salty waters, so the instrument compensates the apparent percent saturation for changes in temperature and conductivity. Most probes measure the current resulting from the electrochemical reduction of oxygen (at a gold cathode) diffusing through a selective membrane. Because oxygen is being removed from the sample to perform the measurement, sample flow is required to prevent false low readings due to depletion of oxygen in the solution in front of the probe. Optical DO probes do not remove oxygen from the sample and are less affected by salts. The common range of DO in groundwater is 0.0 to 3.0 mg/L. Measurements outside of this range suggest that the meter may not be operating correctly.

3.4 **Nephelometric Turbidity Unit (NTU):** The measurement of light passing through a sample based on the scattering of light caused by suspended particles.

3.5 **Potential of Hydrogen (pH):** A measure of acidity and alkalinity of a solution using a logarithmic scale on which a value of 7 represents neutrality, lower numbers indicate increasing acidity, and higher numbers are increasingly basic.

3.6 **Oxidation-Reduction Potential (ORP):** Also known as redox or eH, ORP is a measurement of the potential for a reaction to occur, which generally indicates the oxygen status of a sample. The probe consists of a platinum electrode, the potential of which is measured with respect to a reference electrode that rapidly equilibrates with the potential of the sample solution. A positive value indicates that oxygen is present. A negative value indicates an anaerobic environment or reducing condition. For this reason, negative ORP readings should be associated with DO readings of less than 0.5 mg/l; with negative ORP readings the water may exhibit a sulfur odor or gray color. Positive ORP readings should be associated with DO readings greater than 0.5 mg/L and lack of sulfur odors. Because of the complex relationship between ORP and temperature, no compensation is attempted; it is thus best to report both the ORP and temperature of a water sample.

3.7 **Total Dissolved Solids:** A measure of the quantity of materials in water that are either dissolved or too small to be filtered.

3.8 **Turbidity:** Measure of the clarity of water in NTUs. Potable water typically has NTU values between 0.0 and 0.3 NTUs, depending on the state or regulatory program.

4.0 Interferences

4.1 During field testing, water quality data that is documented from field testing equipment may be influenced by certain outside factors that are unrelated to the actual site water quality. Such parameters and equipment include the following:

4.2 **pH Meters:** Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use isopropyl alcohol very sparingly so that the electronic surface is not damaged.

Poorly buffered solutions with low specific conductance (less than 200 microsiemens per centimeter) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

4.3 **Dissolved Oxygen**: Dissolved gases (e.g., hydrogen sulfide, halogens, sulfur dioxide) are a factor with the performance of DO probes. The effect is less pronounced on optical DO meters. Meter type and potential interferences should be considered based on potential sulfate/sulfide or nitrate/nitrite reducing environments.

Exposure of the sample to the atmosphere will cause elevated DO measurements.

4.4 **Turbidity Meter**: If the weather is warm and humidity is high, condensation may collect on the cuvet.

To avoid this, allow the sample to warm and dry the outside of the cuvet before making the measurement. One method used to accomplish this is to place the cuvet against one’s body (armpits work well).

4.5 **Temperature**: Sample temperature will change rapidly when there are significant differences between the sample and ambient air.

5.0 Training and Qualifications

5.1 Qualifications and Training

5.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

5.2 **Responsibilities**

5.2.1 The TO Manager is responsible for ensuring that field activities comply with this procedure. The TO Manager or designee shall review all surface water sampling forms on a minimum monthly basis. The TO Manager is responsible for ensuring that all field sampling personnel involved in water quality parameter testing shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.

5.2.3 The Field Manager is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 Field sampling personnel are responsible for the implementation of this procedure. Minimum qualifications for field sampling personnel require that one individual on the field team shall have a minimum of 6 months of experience with water quality parameter testing.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the surface water sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling. If deviations from the procedure are

required because of anomalous field conditions, they must first be approved by the Program Quality Manager and then documented in the field logbook and associated report or equivalent document.

6.0 Equipment and Supplies

6.1 The following equipment list contains materials that may be needed in carrying out the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- QAPP;
- Maps/Plot plan;
- Non-water-repellent logbook;
- Site description forms;
- Pump (Peristaltic, Portable Bladder, Submersible);
- Polyethylene bladders (for portable bladder pumps);
- Bladder pump controller (for portable bladder pumps);
- Air compressor (for portable bladder pumps);
- Nitrogen cylinders (for portable bladder pumps);
- 12-volt power source;
- Polyethylene inlet and discharge tubing;
- Silicone tubing appropriate for peristaltic pump head;
- HDPE bailer appropriately sized for well;
- Disposable bailer string (polypropylene);
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity;
- Turbidity meter;
- Teflon-free water level meter; and
- Oil/water interface probe.

6.2 **Equipment/Apparatus**: Field personnel shall consult the site QAPP to review the equipment requirements for the sampling procedures to be followed during the sampling effort. The specific apparatus and materials required will depend on the water quality parameters being monitored. **Table 1** shows the common equipment used in water quality parameter testing.

Table 1
Water Quality Parameter Testing — Common Equipment

Water quality Parameter Instrument	Calibration Standards Required	Other Equipment
pH Meter	Yes - 2- or 3-Point Standards depending on groundwater range. Calibration must cover the range to be measured. If samples are above or below typical buffer standards (4, 7 and 10), special order buffers that fall outside groundwater pH range.	Container or flow thru cell for holding sample
Specific Conductance	Yes	Container or flow thru cell for holding sample
ORP Meter	Yes	Container or flow thru cell for holding sample
Turbidity Meter	Yes	Container or flow thru cell for holding sample
DO	No	Container or flow thru cell for holding sample
Thermometer	No	Container or flow thru cell for holding sample
Flow Rate	No	Container or flow thru cell for holding sample

Notes:
ORP = Oxidation Reduction Potential
DO = Dissolved Oxygen

7.0 Calibration or Standardization

7.1 Instrument or Method Calibration

Most monitoring instruments require calibration before use, and this calibration must be conducted in the field under the ambient climatic conditions that will be present during field sampling. Calibration of monitoring instruments shall be performed in accordance with the manufacturer's specifications and recorded in the provided form in Attachment 1. Site-specific instrument calibration requirements should be specified in the QAPP. The following minimum calibration requirements apply to the various types of meters used to gather water quality measurements.

Initial Calibration (IC): Before use, the instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., DO saturation) or a known value of a calibration standard. An IC is performed in preparation for the first use of an instrument or if a calibration verification does not meet acceptance criteria.

Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following IC by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

Continuing Calibration Verification (CCV): After use, the instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter.

7.2 **Calibration Checks**

Calibration checks are conducted by measuring a known standard. They must be completed after calibration and should be performed at least one other time (i.e., after lunch) and anytime suspect measurements are encountered. Table 2 provides general acceptance ranges to be used during calibration checks. If a meter is found to be outside of the acceptance range, the meter must be recalibrated. If the meter remains out of range, the project manager and/or the supplier of the meter should be contacted to determine alternative measures.

Table 2
Calibration Check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
Mg/L = milligrams per liter
Mv = millivolts
NTU = nephelometric turbidity units

7.3 Possible and Suspected Ranges

The concentration for each parameter range should be known so that concentrations outside of the range can be noted. Table 3 presents the maximum range of the parameter in groundwater. The table also presents the suspected range. Measurements outside of the maximum/minimum range should be considered in error and the measurement method should be checked. Concentrations outside the normal range should be treated as suspect but may be the result of contaminant impact. For example, a pH of 2.0 would be out of the normally suspected range for groundwater but not at a site impacted with an acid.

Table 3
Minimum and Maximum Result Ranges

Parameter	Units	PossibleMin	Possible Max	Normal Min	Normal Max	Notes
Dissolved Oxygen	mg/L	0.0	14.6 (0°C) 10.1 (15°C) 8.3 (2°C)	0.0	5	The colder the sample, the higher the DO reading. DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
pH	SU	0	14	5	9	pH values exceeding 10 could indicate grout contamination

Parameter	Units	Possible Min	Possible Max	Normal Min	Normal Max	Notes
ORP	mv					DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color. DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
Specific Conductance	µS/cm			varies	varies	
Temperature	°C	0	100	5	30	
Turbidity	NTU	0	Greater than 1,000	0	Greater than 1,000	50 NTU or greater suggests cloudiness.

Notes:
mg/L = milligrams per liter
°C = degrees Celsius
DO = dissolved oxygen
SU = standard units
ORP = oxidation reduction potential
Mv = millivolts
mS/cm =micro Siemens per cm
NTU = nephelometric turbidity units

7.4 Field Instruments and Calibration Criteria

The calibration acceptance criteria for each instrument are summarized in Table 4 along with special considerations related to each field instrument.

Table 4
Calibration check Acceptance Limits

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature.
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

Notes:
Mg/L = milligrams per liter
mv = millivolts
NTU = nephelometric turbidity units

7.4.1 pH Meters

For the most accurate of pH measurements, pH meters should receive a three-point calibration. However, if a two-point calibration will bracket the groundwater pH of the site, a two-point calibration is acceptable. Three-point calibrations typically include calibrating to solutions of pH 7.00, 4.00, and 10.00. If groundwater pH is outside the calibration range of the solution standards, special buffers must be ordered to bracket the pH. Some meters will report the slope of the calibration and this may be used in checking the meter calibration (refer to the meter's manual). When performing an ICV, the result must be within +/- 0.2 pH units of the stated buffer value.

pH meters should be calibrated across the range of values to be measured. The maximum and minimum calibration solutions shall be outside the range of anticipated values. For example, if the expected range is between 7.50 and 9.00, the 7.00 and the 10.00 standard should be used for calibration. Perform the IC using at least two buffers, and always use the pH 7.00 buffer first. A reading that is above the maximum (or below the minimum) calibration standard is an estimate only and is not valid. This condition requires obtaining a new standard that is above (or below) the reported value, depending on the measurement.

A percent slope of less than 90 percent indicates a bad electrode that must be changed or repaired. If percent slope cannot be determined, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

7.4.2 Specific Conductivity Meters

For IC, when the sample measurements are expected to be 00 microsiemens per centimeter (µS/cm) or greater, use two standard potassium chloride (KCl) solutions that bracket the range of expected sample conductivities. Calibrate the instrument with the first standard. Verify the calibration of the instrument with the second standard, bracketing the range of expected sample values.

If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values.

When the sample measurements are expected to be less than 100 µS/cm, a lower bracket is not required, but one standard (KCl) solution that is within the range of expected measurements must be used for the IC and the ICV.

Accept the calibration if the meter reads within +/- 5 percent of the value of any calibration standard used to verify the calibration.

Most field instruments read conductivity directly. Record all readings and calculations in the calibration records.

For CCV, check the meter with at least one KCl standard with a specific conductance in the range of conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5 percent of the standard value.

If new environmental samples are encountered outside the range of the IC, verify the instrument calibration with two standards bracketing the range of sample values. If these calibration verifications fail, recalibrate the instrument.

7.5 Dissolved Oxygen Meters

Before calibrating, check the probe membrane for bubbles, tears, or wrinkles. These conditions require replacement of the membrane in accordance with the manufacturer's directions.

If the meter provides readings that are off-scale, will not calibrate, or drift, check the leads, contacts, etc., for corrosion and/or short circuits. These conditions require replacement maintenance in accordance with the manufacturer's directions.

Most DO meters must be calibrated based on an environment of 100 percent humidity and a known elevation and barometric pressure (BP).

For 100 percent humidity, place the probe in the calibration container with a moist towel and allow the probe to remain, undisturbed, for 10 to 20 minutes.

The IC is an air calibration at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument. Allow an appropriate warm up period before IC. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into

the chamber (this ensures 100 percent humidity). Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine from the DO versus temperature table (see Attachment 2) what DO should measure. The acceptance criterion for DO ICV is +/- 0.3 mg/L.

Use the same procedure as above for CCV.

7.6 ORP Meters

Verify electrode response before use in the field.

Equilibrate the standard solution to the temperature of the sample. The standard solution is based on a 25°C temperature; however, the calibration solution standard's value will require adjustment based on the temperature.

Immerse the electrodes and gently stir the standard solution in a beaker (or flow cell).

Turn the meter on, placing the function switch in the millivolt (mv) mode.

Let the electrode equilibrate and record the reading to the nearest millivolt. The reading must be within ±10 mv from the theoretical redox standard value at that temperature. If not, determine the problem and correct it before proceeding. Switch to temperature display and read the value.

Record the mv reading and temperature in the field notebook or in form. Rinse the electrode with distilled water and proceed with the sample measurement, unless using a flow cell. If a flow cell is used, rinse between sample locations.

7.7 Turbidity Meters

Perform an initial calibration using at least two primary standards.

If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard.

Perform an ICV by reading at least one primary standard as a sample. The acceptance criterion for the ICV depends on the range of turbidity of the standard value:

1. Standard Value = 0.1 to 10 NTU: the response must be within 10 percent of the standard;
2. Standard Value = 11 to 40 NTU: the response must be within 8 percent of the standard;
3. Standard Value = 41 to 100 NTU: the response must be within 6.5 percent of the standard; and
4. Standard Value greater than 100 NTU: the response must be within 5 percent of the standard.

Determining the Values of Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards may be used for CCVs.

To initially determine the value of a secondary standard, assign the value that is determined immediately after an ICV or verification with primary standards. This is done by reading the secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10 percent of the assigned standard value. If the +/- 10 percent criterion is not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

CCV: Perform a CCV using at least one primary or secondary standard. The calibration acceptance criteria are the same as those for an ICV.

8.0 Procedures

8.1 Purpose

The procedures will vary depending on parameters being measured, method of sampling, and the method of measurement used. The information here is a general guidance and the site-specific documents and manufacturer manuals supersede these procedures.

8.2 Cautions

Improper use of water quality testing equipment could result in equipment damage or compromised sampling results. Personnel should be trained to operate the test equipment being used for a field operation and should be trained in the proper techniques for collecting and logging water quality parameters. Personnel should also be able to recognize problems with test equipment and have someone available for basic troubleshooting and repair.

8.3 Direct Measurements

Direct measurements with meters are the most common methods and can be accomplished by placing a sample in a container with the probe or by allowing the water to flow past the probe in a flow cell. The use of a flow-through cell improves measurement quality by allowing the constant flow of water over the probes and reduces interaction of the sample with the atmosphere. Sample cups should be avoided. The quantity of samples, timing, and methodology should be described in the project QAPP.

Following calibration of required probes, connect the bottom flow-cell port to the discharge line of the pump. Connect the top port to a discharge line directed to a bucket to collect the purge water. Allow the flow cell to completely fill. As the water flows over the probe, record the measurements. Continue to record the measurements at regular intervals, as specified in the QAPP.

When the ambient air temperatures are much higher or lower than the temperature of the water sample, it is best to keep the length of tubing between the wellhead and the flow cell as short as possible to prevent heating or cooling of the water. Tubing and flow-through cell should not be exposed to direct sunlight, particularly in the summer, if at all possible, to avoid heating of water samples.

8.4 Data Acquisitions, Calculations, and Data Reduction

8.4.1 Specific Conductivity Correction Factions

If the meter does not automatically correct for temperature (i.e., read Specific Conductivity) record Conductivity and adjust for temperature upon returning to the office. The following equation can be used to convert Conductivity to Specific Conductivity.

$$K = \frac{(Km)(C))}{1 + 0.0191(\mathrm{T} - 25)}$$

Where:

K = Conductivity in µmhos/cm at 25°C

Km = Measured conductivity in µmhos/cm at T degrees Celsius

C = Cell constant

T = Measured temperature of the sample in degrees Celsius;

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(Km)}{1 + 0.0191(\mathrm{T} - 25)}$$

8.4.2 Percentage Difference Calculation

For evaluating slope of readings from either a flow cell or a sample cup.

$$\%Difference = \frac{(Highest\ Value - Lowest\ Value)}{(Highest\ Value)}\ x\ 100$$

8.4.3 Convert mm mercury (mmHG) to inches mercury (inHG)

$$mmHG = inHG\ x\ 25.4$$

8.4.4 True Barometric Pressure

For Converting BP obtained from a public domain source that is expressed in BP at sea level to BP at the subject site.

$$TrueBP = (BP) - \frac{(2.5\ x\ [Local\ Altitude])}{100}$$

Where: BP is in mmHG and Local Altitude is in feet

Example: BP at Site A is 30.49 inHG and elevation is 544 feet, calculate TrueBP

Convert inHG to mmgHG:

mmHG = 30.49 inHG x 25.4 = 774.4 mmHG

Calculate TrueBP:

TrueBP = (774.4 mmHG) – [2.5*(544/100)] = 774.4-13.6 = 760.mmHG.

9.0 Quality Control and Assurance

9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific QAPP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.

9.2 Quality Control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific QAPP will provide requirements for sample preservation, holding times, container types, as well as various QC samples such as trip blanks, field blanks, equipment blanks, and field duplicates.

10.0 Data and Records Management

10.1 Field notes will be kept during sampling activities in accordance with *SOP 3-03 – Recordkeeping, Sample Labeling, and Chain of Custody*. During the completion of sampling activities, fill out the sample logbook and transmit forms to the TO Manager for storage in project files.

10.2 Deviations to the procedures detailed in the SOP should be recorded in the field logbook.

11.0 Attachments or References

Attachment 1: Example Field Instrument Calibration Form
Attachment 2: Solubility of Oxygen at Given Temperatures
Attachment 3: Example Field Data Form

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Naomi Ouellette, Project Manager	Rev 0 – Initial Issue
Amanda Martin Engineer	Claire Mitchell, PE, PMP Senior Engineer	Rev 1 – PFAS sampling update (July 2019)
Rose Kelley Environmental Scientist	Richard Purdy Project Scientist	Rev 2 – Update & Review (June 2022)

Attachment 1
Example Field Instrument Calibration Form

EQUIPMENT CALIBRATION DAILY LOG	
Date:	Project Name:
Project Number:	Recorded By:

					Morning Calibration	Evening Check	Additional Calib./Check (if necessary)
PID	Model:		Bulb:				
	Equipment ID #:						
	Parameter	Standard	Exp. Date	Lot #	Time:	Time:	Time:
First Point Calibration	Vapor conc. (ppm)	0.0 (ambient air)	NA	NA	Initials:	Value:	
Second Point Calibration	Vapor conc. (ppm)	100 (isobutylene)			Initials:	Value:	

					Morning Calibration	Evening Check	Additional Calib./Check (if necessary)
COMB. GAS/O_2 METER	Model:		Bulb:				
	Equipment ID #:						
	Parameter	Standard	Exp. Date	Lot #	Time:	Time:	Time:
First Point Calibration	O_2 (%)	20.9%			Initials:	Value:	
	H_2S (%)	25 ppm			Initials:	Value:	
	CO (%)	50 ppm			Initials:	Value:	
	% LEL Pentane	50% (methane)			Initials:	Value:	

					Morning Calibration/Check	Evening Check (one point only)	Additional Calib./Check (if necessary)
WATER QUALITY METER	Model:						
	Equipment ID #:						
	Parameter	Standard	Exp. Date	Lot #	Time:	Time:	Time:
First Point Calibration (Auto)	pH	4.00			Initials:	Value:	
	Conductivity (mS/cm)	4.49				Value:	
	Turbidity (NTU)	0				Value:	
	DO (mg/L)	8.9-9.1 (ambient air)	NA	NA		Value:	
Second Point Calibration	pH	7.0			Initials:	Value:	
	Conductivity (mS/cm)					Value:	
	Turbidity (NTU)	100				Value:	
Third Point Calibration	pH	10.0			Value:	Value:	
	Conductivity (mS/cm)				Value:	Value:	
	Turbidity (NTU)				Value:	Value:	

Additional Remarks:

Attachment 2
Solubility of Oxygen at Given Temperatures

Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water at Atmospheric Pressure			
Temperature	**Oxygen Solubility**	**Temperature**	**Oxygen Solubility**
°C	**mg/L**	**°C**	**mg/L**
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

Notes:
The table provides three decimals to aid interpolation
Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water saturated
°C = degrees Celsius
mg/L = milligrams per liter

Attachment 3
Example Field Data Form

AECOM

Monitoring Well Sample Collection Form

Page 1 of ____

LOCATION	Site:	LocID:	Date:
	Project Name:	Project Number:	Recorded By: Checked By:
EQUIPMENT	Sampling Equipment - Pump:	Controller:	Compressor:
	Water Level Indicator Type/ID#:	Water Quality Meter Type: Sonde ID:	Handset ID:
	PID Type/ID#:	Equipment Decon:	
WELL & SAMPLING INFO	Description:	Screen Interval (BTOC):	Initial Depth to Water (BTOC): / Ambient PID (ppm):
	Historic Pump Settings:		Pump Inlet Depth (BTOC): / Well Head PID (ppm):
	Condition of Well/Comments:		
	NOTE:		

Date (MM/DD/YY)	Time (24 hr)	Depth to Water (BTOC)	Volume Removed (gallons)	Pumping Rate (Lpm)	Temp (°C)	Specific Conductivity (mS/cm)	DO (mg/L)	pH	ORP (mV)	Turbidity (NTU)	Pump Refill/ Discharge (seconds)	Pump Pressure (PSI)	Comment

Pumping Rate: ≤ 0.5L/min; **Measurements**: every 3 - 5 minutes; **Stabilization is defined as the following for three consecutive readings**: ± 3% Temp, ± 3% Conductivity; + 10% DO; ± 0.1 pH; ± 10mV ORP; 10% Turb

Sample ID Numbers and Sample Time	Container Count, Volume & Type	Preservative	Parameter(s)

Appendix B: Laboratory Certificates

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: _____ Date: _____

Approved By:

Department Manager: _____ Date: _____

Operations Manager: _____ Date: _____

QA Officer: _____ Date: _____

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	updated sect 5.0 with current spk slolutions, removed medium level extraction procedure, updated fig 2 &3 (lcs/ms spike components), minor corrections			
05	Many changes made through out, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed with SOP in QA for a detailed list of changes.			

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook page. Added adipate compounds to Fig. 2. Added the necessity of recording lot numbers of consumables in the extraction logbook.			
07	Added requirement to add spike before NaSO4. Changed N2 water bath temperature from < 39°C to 30 °C. Removed respirator references. Added KAEHS manual. Updated to SW846 3550C. Added KA SOP CA-108 for additional subsampling information.			
08	Removed targeting sample weights. Added reference KA SOP SD-902 reference. Updated logbook page. Added GPC cleanup is required for all samples. Removed to decant samples.			
09	Minor modifications made to sections 5 and 7 to reflect current practices. Updated section 9.0 to include LOD/LOQ requirements. Changed 7.6 and 7.7 to add surrogate and spikes after sodium sulfate is added. Updated references section 10.0.			
10	Sect 5 – Updated surrogate preparation for both SIM and Scan surrogates in 1 mix. Sect. 7 – Updated spiking information for SIM/Scan surrogate mix. Clarified decanting samples. Sect. 10 – Added and updated references. Updated Figure 1.			
11	Sect. 4 and 7 – Updated for new sonicator. Changed KAS INC to KAS throughout			
12	Title changed for sections 1.4 and 5.0. Updated method references for NELAC, DoD and SW846. Removed old sonicator parameters. Removed a duplicate paragraph. Clarified sample weight.			
13	Sect. 7 – Updated M.BLK and LCS initial weight criteria. Sect. 10 – Updated references. Updated logbook example.			

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Added use of tongue depressors. Corrected typographical errors.	Lesia Dimond	04/19	04/19
15	Sect. 4 and 7 – Added 8 oz. soil jars as extraction vessels. Sect. 2, 4 and 7 – Added summary, materials and procedure for high concentration extraction.	Lesia Dimond	07/20	07/20

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-15**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-15**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550C in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

For low concentration soils, a 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic 3/4" horn. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

For high concentration soils, a 2 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an 1/8 inch tapered microtip attached to a 1/2-inch ultrasonic horn. The methylene chloride extract is dried and concentrated to a volume of 0.5 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Pasteur pipets - disposable, 5 ¾ ".

4.3 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.4 Sonicator ultrasonic processor XL – QSonica Model Q500 (or equivalent)

- equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.
- A 1/8 inch tapered microtip attached to a 1/2-inch horn for the medium/high concentration method procedure

4.5 Vacuum filtration flask - 500 mL Erlenmeyer

4.6 Filter paper, 70 mm, Whatman #4

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 4oz. clear soil jars for extraction of low concentration samples

4.9 20 mL vial for extraction of high concentration samples.

4.10 Spatula - stainless steel

4.11 Wooden Tongue Depressors

4.12 Balance - capable of accurately weighing ± 0.01 g.

4.13 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent).

4.14 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.15 Powder funnels, 100 mm diameter, 35 mm stem

4.16 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.17 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.18 Nitrogen evaporation apparatus.

4.19 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.20 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 200 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 SCAN/SIM Surrogate Spiking Solution - A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.7 Base/Neutral and Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2.0 ug/mL for base/neutral and 4.0 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Sonicator horns tuned
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples should be cleaned using gel permeation chromatography (GPC) to reduce matrix interferences.

The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP SD-902, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples. Fill out the sample preparation/extraction log with the necessary information before starting the extraction. Pre-rinse all glassware three times with methylene chloride.

EXTRACTION OF LOW LEVEL EXTRACTS (<20 mg/Kg)

7.1 Do not decant any water on the sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2 Mix with a stainless-steel spatula or wooden tongue depressor to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic

Laboratory Technique ", for more detailed guidance on subsampling to ensure reproducibility.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out an approximate, greater than 30 g portion of sample into a labeled 4oz. soil jar. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula or wooden tongue depressor. Keep the spatula or wooden tongue depressor in the sample container and cover the container with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 $\pm$ 0.05 g portion of purified sand in a labeled 4oz soil jar. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ± 0.05 g portion of purified sand in a labeled 4oz soil jar. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 4oz soil jars. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **after** the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook.

Thoroughly rinse syringe with solvent prior to using it for another spiking solution. **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **after** the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 Turn sonicator on

7.9.1 Set pulse timer to 1 ½ minutes.

7.9.2 Set pulse to 1 and 1. This sets the sonicator to pulse for 1 second and rest for 1 second, with the timer set for 1 ½ minutes this equals a 3 minute total sonication

7.9.3 Set amplitude to 40%. Record this in the logbook.

7.9.4 These settings are stored in the unit and do not have to be entered with each use.

7.9.5 When done sonicating turn off the unit.

7.9.6 Refer to the Operating Manual for further information.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add approximately 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula or wooden tongue depressor to loosen up the mixture prior to extracting. Rinse the spatula or wooden tongue depressor with methylene chloride and collect the rinsing into a correspondent 4oz soil jar. Position the 4oz soil jar in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the 4oz soil jar under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written in the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using approximately 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula or wooden tongue depressor. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

MEDIUM/HIGH CONCENTRATION EXTRACTION PROCEDURE (>20 mg/Kg)

7.16 Transfer approximately 2 g of sample to a 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination. Record the weight to the nearest 0.1 g.

7.17 For the sample in each batch selected for spiking, add 1.0 mL of the matrix spiking solution.

7.18 Add 1.0 mL of surrogate spiking solution to all samples, spiked samples, QC samples, and blanks.

7.19 Nonporous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 2 g of anhydrous sodium sulfate, using a spatula.

If needed, more sodium sulfate may be added. After addition of sodium sulfate, the sample should be free flowing

7.20 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0 mL, considering the added volume of surrogates and matrix spikes.

7.21 Extract the sample with the 1/8-inch tapered microtip ultrasonic probe for 2 min at output control setting 5 and with mode switch on pulse and percent duty cycle at 50%.

7.22 Loosely pack a disposable Pasteur pipette with 2 to 3 cm of glass wool. Filter the sample extract through the glass wool and collect the extract in a suitable container.

7.23 The entire 10 mL of extraction solvent cannot be recovered from the sample. Therefore, the analyst should collect 5.0 mL of extract in a clean concentrator tube. This volume represents exactly half of the total volume of the original sample extract and will need to be concentrated to a final volume of 0.5 mL. Proceed to section 7.30 for the nitrogen blowdown procedure.

CONCENTRATION OF EXTRACTS

7.24 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written in the extraction logbook.

7.25 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.26 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.27 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.28 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.29 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.30 Reduce the extract in the concentrator tube to approximately 1 mL (0.5 mL for high concentration soils) using the nitrogen blow-down apparatus. The bath temperature must be < 39°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.31 When the apparent volume reaches slightly less than 1 mL (0.5 mL for high concentration soils), remove the concentrator tube and allow it to cool.

7.32 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL (0.5 mL for high concentration soils) using the 1.8 mL reference vial for volume comparison.

7.33 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and “tray location” of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

A method blank must be extracted for each item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases, data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client, and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3550C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The 2009 TNI Standards

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

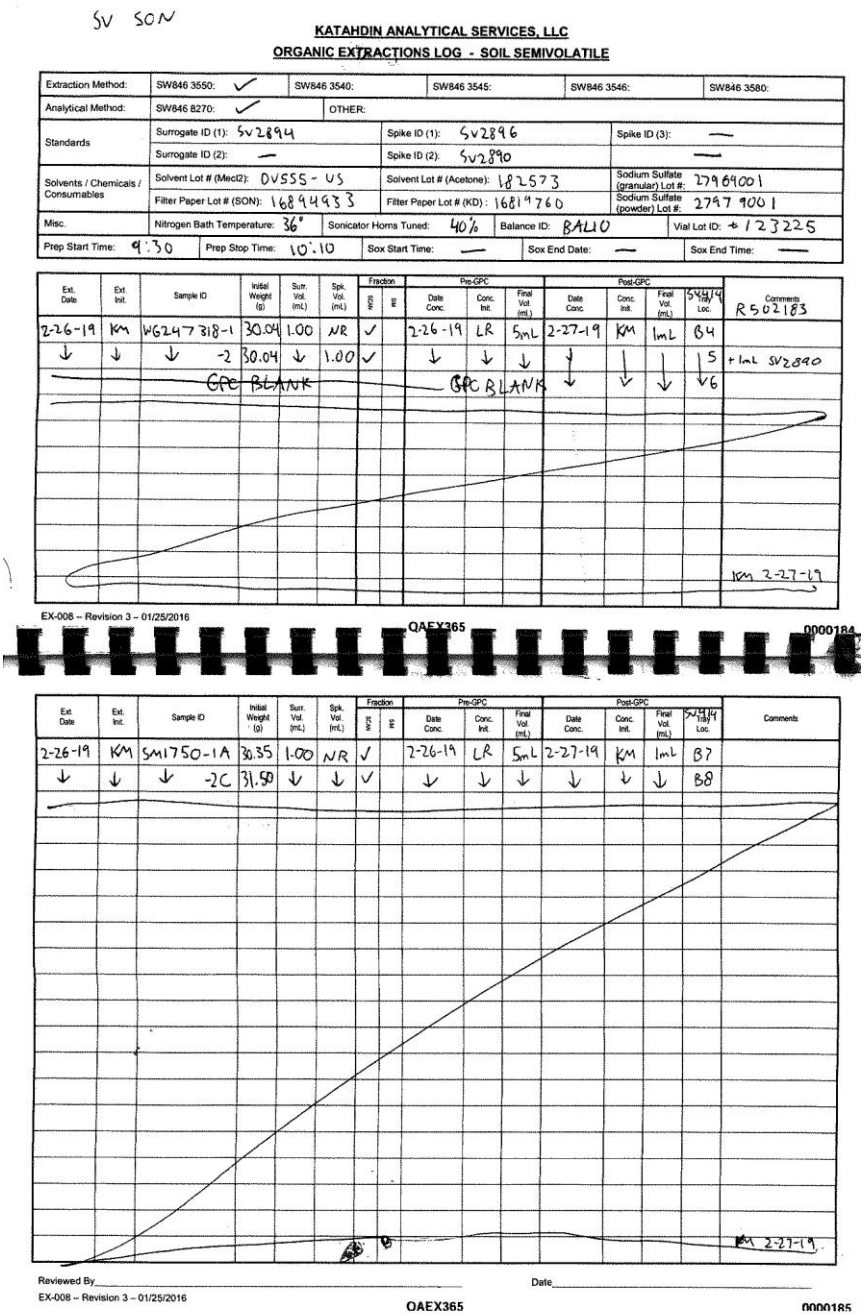
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-15	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV SON

KATAHDIN ANALYTICAL SERVICES, LLC

ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV2894		Spike ID (1): SV2896	Spike ID (3): —	
	Surrogate ID (2): —		Spike ID (2): SV2890	—	
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): DV555-US		Solvent Lot # (Acetone): 182573	Sodium Sulfate (granular) Lot #: 27989001	
	Filter Paper Lot # (SON): 16894933		Filter Paper Lot # (KD): 16819760	Sodium Sulfate (powder) Lot #: 2797 9001	
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: 40%	Balance ID: BAL10	Vial Lot ID: # 123225	
Prep Start Time: 9:30	Prep Stop Time: 10:10	Sox Start Time: —	Sox End Date: —	Sox End Time: —	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WDS	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SW846 Tray Loc.	Comments
2-26-19	KM	WG247318-1	30.04	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B4	R502183
↓	↓	↓ -2	30.04	↓	1.00	✓		↓	↓	↓	↓	↓	↓	5	+1mL SV2890
		GPC BLANK							GPC BLANK		↓	↓	↓	V6	
															KM 2-27-19

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WDS	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	SW846 Tray Loc.	Comments
2-26-19	KM	SM1750-1A	30.35	1.00	NR	✓		2-26-19	LR	5mL	2-27-19	KM	1mL	B7	
↓	↓	↓ -2C	31.50	↓	↓	✓		↓	↓	↓	↓	↓	↓	B8	
															KM 2-27-19

Reviewed By_____ Date_____

EX-008 – Revision 3 – 01/25/2016 OAEX365 0000185

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: J Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.16.00

General Manager: Dennis F. Hoffman Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~spikes~~ compounds used. Updated sect. 5.0 to include all compounds analyzed for. Updated logbook page. Minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1 Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added acidic compounds to Fig. 2. Added recording of consumable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09
07	Removed targeting sample weights. Added to weigh out a minimum 30g sample. Removed decanting samples prior to extracting. Added putting solid waste in the "I" waste stream. Updated Logbook page.	LAD	08/10	08/10
08	Section 5.5 - Added 1,4-Dioxane-D8 and updated 2,4-Dibromophenol's concentration. Section 5.7 - Updated acid spikes concentration. Sections 7.0, 7.6 and 7.13 - Minor changes to reflect current practice. Added MDL, LOD and LOQ information to Section 9. Updated references in Section 10.	LAD	04/12	04/12
09	Sect. 5 - Updated Surr. prep. for both SIM and Scan Surr. - now in 1 mix. Sect. 7. Updated spiking info. for SCAN/SIM surr. Clarified decanting soils. Sect. 10 - Added and updated references. Updated Fig. 1.	LAD	06/14	06/14
10	Sect. 1 - Added pollution control to Waste Disposal Sect. 5 - Added standards to title. Sect. 7. Added to record the date Sox. ends, changed M. BIK and LCS initial weigh from >30g to 30g. Updated Fig. 1 - logbook ex. KAS INC → KAS throughout	LAD	08/16	08/16
11	Updated method references for DOD+SW846. Clarified weight of sample. Fixed grammatical errors. Changed water bath temperature.	LAD	09/17	09/17
12	Added wooden tongue depressors, updated Rheostat setting, updated references. Corrected typographical errors	LAD	04/19	04/19
	LAD 082820			

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
13	Sect. 5.3 – Added reagent number needed for baked sand. Sect. 7 – Added to record the reagent number in the logbook. Updated references.	Leslie Dimond	08/20	08/20

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest

the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

4.1.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.2 500 mL flat-bottom boiling flask

4.1.3 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Wooden Tongue Depressors

4.17 Long forceps, stainless-steel

4.18 Metal clips – for securing Soxhlets to boiling flasks

4.19 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS AND STANDARDS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, pre-baked, certified by the manufacturer/vendor.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand. Record prep in Reagent Logbook and give a unique ID.

5.4 SV SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.6 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral and 4 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers/Reagent numbers of all solvents, acids and bases, sodium sulfate, filter paper, baked sand.
- Nitrogen evaporation water bath temperature
- pH
- Extraction and concentration dates
- Extraction and concentration analyst
- Soxhlet extraction start and end dates and times
- Prep Date and start and end times.
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

Fill out the sample preparation/extraction log with the necessary information before starting the extraction.

Pre-rinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Do not decant any water layer on a sediment sample.

Note: Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

7.2.2 Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, "Basic Laboratory Technique" for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out an approximate, greater than 30 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30 g ±0.5g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g ±0.5 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the SV SCAN/SIM surrogate spiking solution using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 40-45% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as much solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures along with the glass wool from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries. Put the solid waste in the "I" waste stream.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate

crystals. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As

the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases, data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ

must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 3540C.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

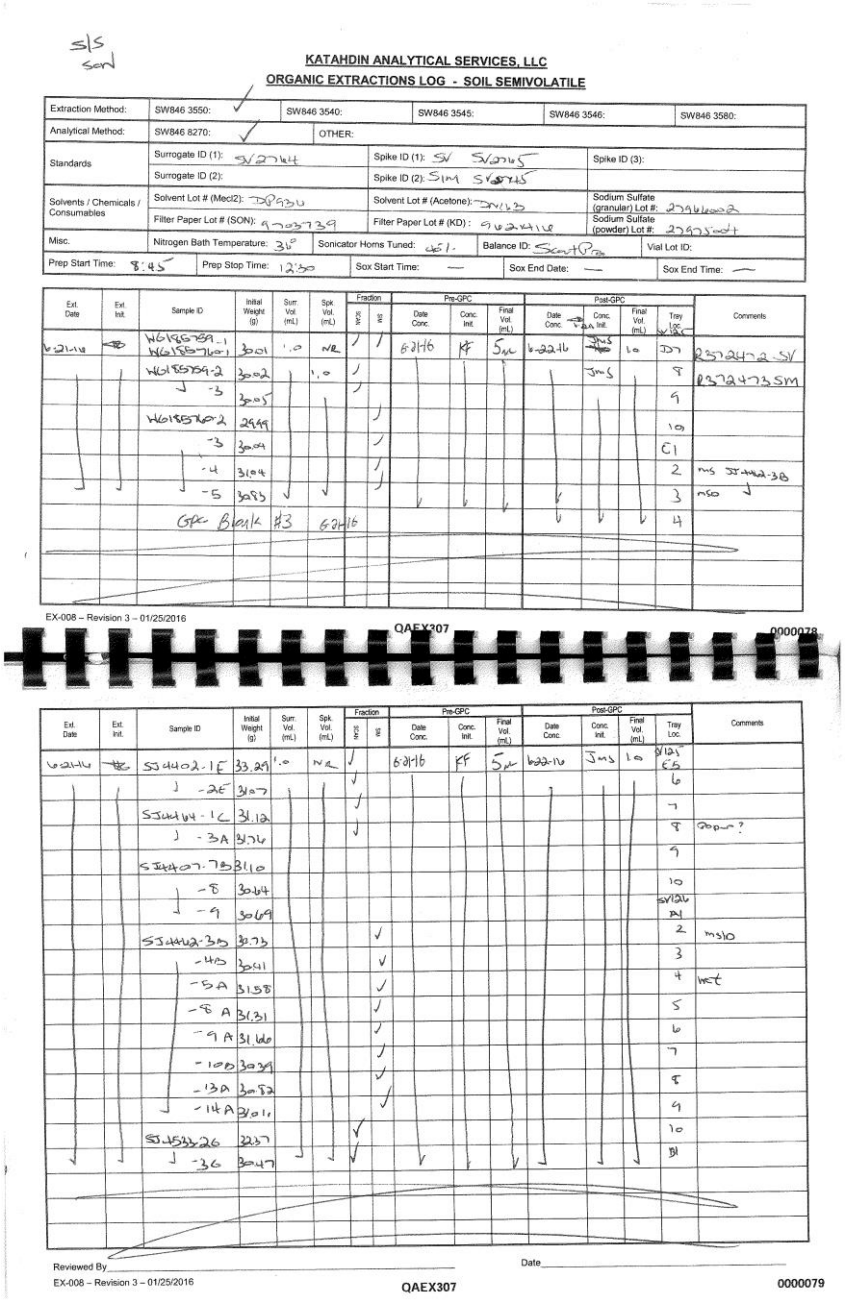
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-13	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES, LLC
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method:	SW846 3550: ✓	SW846 3540:	SW846 3545:	SW846 3546:	SW846 3580:
Analytical Method:	SW846 8270: ✓	OTHER:			
Standards	Surrogate ID (1): SV2764	Spike ID (1): SV SV0765	Spike ID (3):		
	Surrogate ID (2):	Spike ID (2): SIM SV0715			
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): 169936	Solvent Lot # (Acetone): DN1623	Sodium Sulfate (granular) Lot #: 279616002		
	Filter Paper Lot # (SON): 9703739	Filter Paper Lot # (KD): 9628411B	Sodium Sulfate (powder) Lot #: 279351mt		
Misc.	Nitrogen Bath Temperature: 36°	Sonicator Horns Tuned: ds/.	Balance ID: Scout Pro	Vial Lot ID:	
Prep Start Time: 8:45	Prep Stop Time: 12:30	Sox Start Time: —	Sox End Date: —	Sox End Time: —	

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WCS	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-21-16		W61855891 W61855901	30.01	1.0	NA	✓	✓	6-21-16	KF	5mL	6-22-16	JMS	1.0	1D7	R3724732 SV
		W61855892	30.02	1.0		✓						JMS		8	R3724733 SM
		-3	30.05			✓								9	
		W61855902	29.99			✓								10	
		-3	30.09			✓								C1	
		-4	31.04			✓								2	ms JJ-MB2-38
		-5	30.93	✓	✓	✓		✓	✓	✓	✓	✓	✓	3	msd
		GPC Blank #3		6-21-16										4	

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Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WCS	Fraction WS	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-21-16		SJ4402-1E	33.29	1.0	NA	✓		6-21-16	KF	5mL	6-22-16	JMS	1.0	SV126 E5	
		-2E	30.07			✓								6	
		SJ4464-1C	31.12			✓								7	
		-3A	31.76			✓								8	dupe?
		SJ4407-7B	31.10			✓								9	
		-8	30.64			✓								10	
		-9	30.69			✓								SV126	
		SJ4442-3B	32.73				✓							2	msd
		-4B	30.41				✓							3	
		-5A	31.58				✓							4	wet
		-8A	31.31				✓							5	
		-9A	31.66				✓							6	
		-10B	30.39				✓							7	
		-13A	30.52				✓							8	
		-14A	31.01				✓							9	
		SJ4532-26	32.37			✓	✓							10	
		-36	30.47	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	B1	

Reviewed By_____ Date_____

EX-008 – Revision 3 – 01/25/2016 QAEX307 0000079

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-604
Revision History
Cover Page
Page 1

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Madeau Date: 1·22·01

General Manager: Duncan F. Heffron Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DH	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DH	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Figures 2 and 3. Changed KAS, INC. to KAS.	LAD	06/15	06/15
07	Sect. 1 - Replaced beaker with digestion vessel. Sect. 7.5 & 7.9 - Added calibrated pipet.	LAD	06/16	06/16
08	Update figure 1. Change title of section 5.0 to Reagents and Standards. Update method references for NELAC and DoD.	LAD	09/17	09/17
09	Added Thorium to Tables 2 and 3 Sect. 8 - Added contingency plan.	LAD	01/19	01/19
10	Added 5000ul pipet to sect. 4.3. Updated Figure 1 to new bench sheet.	LAD	02/21	02/21
11	Sect. 1.4 - Changed digestate storage from 60 days to 90 days. Sect. 7 - Added Block Digestor instructions. Other minor edits to reflect current practices	LAB	03/22	03/22

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

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-604-11**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-11**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever digestion vessels are being heated in the hood. Use caution when handling hot digestion vessels.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 90 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 90 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers. If digestion will be performed using a hot plate, use 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning")..

4.2 Ribbed watch glasses. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used.

4.3 Adjustable volume automatic pipets covering the range from 20 uL to 5000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Block digester, hot plate, or other heating source - adjustable and capable of maintaining a temperature of 90-95^{o}C. Block digesters and hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. For block digesters, a digestion tube containing reagent water in which a thermometer is immersed may be used. When using a hot plate, this may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. The temperature of each digestion block or hot plate in use is measured each day. The block digester/hot plate identification number and the measured temperature are recorded on the sample preparation bench sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place

a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation

date, analyst initials, etc.) into the Metals database bench sheet. Print out a copy of the bench sheet. With a permamament marker, label the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter or calibrated pipet, to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample for a minimum of 8 minutes and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating for 15 minutes.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample for a minimum of 8 minutes and use a repipetter or calibrated pipet to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume

heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool for a minimum of 8 minutes.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the bench sheet for accuracy. If any information is incorrect, make the necessary changes to the bench sheet and print out a corrected copy. Do not discard the original copy of the bench sheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, block digester/hot plate ID and temperature in the appropriate spaces on the bench sheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (bench sheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, January, 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-11	EPA METHOD 3010, current revision
Apparatus/Materials	1) 2) Digestion performed in 70ml digestion tube, or 250 mL/400 mL Griffin beaker to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample for a minimum of 8 minutes and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample for a minimum of 8 minutes and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Reagents and Consumables Information:

HNO3: MSR221 HCL: MSR219

1:1 HNO3: MR2960 1:1 HCL: MR2944

Pipet **LCS/Spiking Information:**

M41 CLPP-SPK-1 (ID/Vol): MS2282 / 0.05 mL

M21 CLPP-SPK-INT1 (ID/Vol): MW19933 / 0.5 mL

M21 CLPP-SPK-INT2 (ID/Vol): MW20001 / 0.5 mL

— — Spike (ID/Vol): — / — mL

Metals Preparation Benchsheet

Method: 3010-MS

Digestion Vessels: MLDO2660000416

Heat Source ID : A

Start Time: 9:03 /Temp. 94 °C

End Time: 1543 /Temp. 90 °C

Thermometer ID/Pos: ALC30 / 1:3

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle	pH <2
LCSWOB04IMW1	OB04IMW1	0.05	L	2.05	L	AQ	IM	SF	02/04/2021	—	✓
PBWOB04IMW1	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	—	
SO0588-001	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	G	
SO0588-002	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	T	
SO0588-002P	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	T	
SO0588-002S	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	T	
SO0588-003	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	G	
SO0588-004	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	G	
SO0597-009	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	D	
SO0597-010	OB04IMW1		L		L	AQ	IM	SF	02/04/2021	D	
SO0597-011	OB04IMW1	✓	L	✓	L	AQ	IM	SF	02/04/2021	D	✓

Client IDs verified -SF

REVIEWED
EP 2/5/21
KATAHDIN ANALYTICAL
METALS SECTION

QA-064-Revision 5 -12/07/2018 Digestion performed by: SF On: 2/4/21 Page: OB013 Revision: 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10,000 mg/L K	High Purity Standards	10.0
	10,000 mg/L Na	High Purity Standards	7.5
	10,000 mg/L Mg	High Purity Standards	5.0
	10,000 mg/L Ca	High Purity Standards	2.5
	1000 mg/L Tl	High Purity Standards	1.0
CLPP-SPK-INT2	1000mg/L Sr	High Purity Standards	5.0
	1000mg/L Sn	High Purity Standards	5.0
	10,000mg/L Si	High Purity Standards	1.0
	1000mg/L B	High Purity Standards	5.0
	1000mg/L Li	High Purity Standards	5.0
	1000mg/L Ti	High Purity Standards	5.0
	1000mg/L Mo	High Purity Standards	1.0
	1000mg/L U	High Purity Standards	1.0
	1000mg/L W	High Purity Standards	1.0
	1000mg/L Th	High Purity Standards	1.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION		
	CLPP-SPK-1	CLPP-SPK-INT1	CLPP-SPK-INT2
Aluminum	2000		
Antimony		10	
Arsenic		10	
Barium	2000		
Beryllium	50		
Boron			50
Cadmium		25	
Calcium		250	
Chromium	200		
Cobalt	500		
Copper	250		
Iron	1000		
Lead		10	
Magnesium		500	
Manganese	500		
Molybdenum			10
Nickel	500		
Potassium		1000	
Selenium		10	
Silicon			100
Silver	50		
Sodium		750	
Strontium			50
Thallium		10	
Tin			50
Titanium			50
Uranium			10
Vanadium	500		
Zinc	500		
Lithium			50
Tungsten			10
Thorium			10

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-605
Revision History
Cover Page
Page 1

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Joel C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Hufford Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables	DH	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DH	8.29.02	8.29.02
03 3050B	New Title to include 1. MOS, 3. Use of digestion block and polyethylene digestion tubes added to sections 9.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 2.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 7 – Added wording concerning subsampling. Table 3 and 4 – Corrected standard concentrations. Attachment A - Modifications For 8330B Preparation & Digestion. Changed KAS INC. to KAS throughout	LAD	08/15	08/15
07	Update Figure 2. Change title of Section 5.0. Update method references for NELAC + DoD. Minor changes to Table 1 + section 8.2.	LAD	09/17	09/17
08	Updated Table 3 to include Thallium and corrected standard concentration of U + W. Updated Table 4 to include Thallium Sect. 8 - added Contingency Plan. Sect 10. updated references	LAD	01/19	01/19
09	Sect. 4 - Added PTFE boiling chips. Sect. 8 - Added PTFE boiling chips to LCS preparation.	LAD	01/20	01/20
10	Sect. 4.13 - Added balance make and model	LAD	06/20	06/20
11	Changed pipet range to 1ml → 5000ul in Sect. 4.3. Updated Fig 1 with new bench sheet.	LAD	02/21	02/21
12	Sect. 14 - Changed retain digestates from 60 days to 90. Sect. 4 - Removed specimen cups and repipeters. Sect. 5 - added option to use Optima grade H_2O_2. Changed Access Spreadsheet to bench sheet throughout	LAD	03/22	03/22

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

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-605-12**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-12**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS.**

Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSO – Laboratory Control Sample for Solids – An aqueous standard that had been brought through the sample preparation process.

LCSS – Laboratory Control Sample for Solids – A solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the

samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from the Environmental Health and Safety Officer, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 90 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 90 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 5000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.5 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recorded each day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.6 Plastic funnels, pre-cleaned as in Section 4.1.

4.7 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.8 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.9 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.10 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.11 Analytical balance capable of reading to 0.01 gram, Ohaus model SPE402, or equivalent.

4.12 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not require to be rinsed.

4.13 Pre-cleaned PTFE boiling chips. These are cleaned by soaking in 5% nitric acid overnight, then rinsing thoroughly with reagent water, and air drying.

5.0 REAGENTS AND STANDARDS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade. Optima grade 30% H_2O_2 may be used if requested.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the preparation logbook sheet (bench sheet). Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Avoid rocks, roots, leaves and other organic or inorganic foreign material. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet.
Refer to Katahdin Analytical Services SOP CA-108, current revision “Basic Laboratory Technique” for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using a pipette, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass or a disposable polyethylene watch glass and place on heat source. Gently heat the sample to 95^{o}C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample for a minimum of 8 minutes.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until two hours have elapsed. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample for a minimum of 8 minutes.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating for 15 minutes.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction for 15 minutes.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming for 15 minutes with 8 minutes of cooling between each aliquot added.

7.12 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^{o}C ± 5^{o}C for 15 minutes. Remove the sample from the heat source and cool.

7.13 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid

container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.14 Review the bench sheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the bench sheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation bench sheet) is included as Figure 2.

7.15 Reopen the electronic bench sheet in the metals database for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.15 Place each batch of digestates in a box labeled with the QC Batch ID and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.16 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new

reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of PTFE boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 Prepare an appropriate number of laboratory control samples. Typically, the laboratory control sample is prepared by weighing 1.0 g of PTFE boiling chips into a digestion vessel and spiking as described in Table 3. This type of laboratory control sample is identified as "LCSO" in sample preparation and analysis records. Alternatively, a laboratory control sample may be prepared by weighing ~~an appropriate amount~~ 1 to 2 grams of a solid reference material into a digestion vessel. Laboratory control samples prepared from solid reference materials are identified as "LCSS". Laboratory control samples are digested using the same reagents as those used to digest associated samples. The analyte concentrations of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense (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.

LIST OF TABLES AND FIGURES

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Aqueous or Solids (LCSO or LCSS)	One each per prep batch of 20 or fewer samples, if specified by project or client	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-12	Method 3050, current revision
Apparatus /Materials	1) 70 mL polyethylene tube. 2) 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	1.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	1.0
	1000 mg/L U	High Purity Standards	1.0
	1000 mg/L W	High Purity Standards	1.0
	1000 mg/L Th	High Purity Standards	1.0

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L			
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**
Aluminum	2.000	2000		
Antimony	0.100		10	
Arsenic	0.100		10	
Barium	2.000	2000		
Beryllium	0.050	50		
Boron	0.500			50
Cadmium	0.250		25	
Calcium	2.500		250	
Chromium	0.200	200		
Cobalt	0.500	500		
Copper	0.250	250		
Iron	1.000	1000		
Lead	0.100		10	
Lithium	0.500			50
Magnesium	5.000		500	
Manganese	0.500	500		
Molybdenum	0.300			10
Nickel	0.500	500		
Potassium	10.000		1000	
Selenium	0.100		10	
Silicon	5.000			100
Silver	0.050	50		
Sodium	7.500		750	
Strontium	0.500			50
Thallium	0.100		10	
Tin	0.500			50
Titanium	0.500			50
Tungsten	0.100			10
Uranium	0.100			10
Vanadium	0.500	500		
Zinc	0.500	500		
Thorium	0.100			10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out bench sheet.

2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.

3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.

4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.

5. Add spike solutions to matrix spike samples.

6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.

7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.

8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.

9. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.

10. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat for 15 minutes and cool for at least 8 minutes.

12. Cool sample and add 2 mL 30% H_2O_2. Heat ~~gently until effervescence subsides~~ for 15 minutes and cool for at least 8 minutes.

13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat for 15 minutes and cool for at least 8 minutes.

15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C and cool for at least 8 minutes.

16. Cool sample. Filter and bring to volume with reagent water ~~and transfer to~~ in a labeled polyethylene bottle.

17. Enter sample weights into bench sheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. **Metals Preparation Benchsheet** **Method:** 3050H-MS

Reagents and Consumables Information:

HNO3: MSR221 HCL: MSR219 Digestion Vessels: MLDD260000446 Filter Paper: 17094497
1:1 HNO3: MR2400 H2O2: MSR223 Boiling Stones: MSR207

Pipet	LCS	Spike	LCS/Spiking Information:
M11	☐	☑	CLPP-SPK-1 (ID/Vol): MS2282 / 0.1 mL
M21	☑	☐	CLPP-SPK-INT1 (ID/Vol): MW19433 / 1.0 mL
M21	☑	☑	CLPP-SPK-INT2 (ID/Vol): MW19434 / 1.0 mL
—	☐	☐	___ Spike (ID/Vol): ___ / ___ mL

LCSS: ___ Balance ID: 09

Heat Source ID: B
Start Time: 8:54 Temp. 94 °C
End Time: 1553 Temp. 75 °C
Thermometer ID/Pos: ALL25 / 2:6

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOOB03IMS1	OB03IMS1	1.0	g	0.1	L	SL	IM	SF	02/03/2021	—
PBSOB03IMS1	OB03IMS1	1	g		L	SL	IM	SF	02/03/2021	—
SO0049-017	OB03IMS1	1.08	g		L	SL	IM	SF	02/03/2021	F
SO0049-018	OB03IMS1	1.04	g		L	SL	IM	SF	02/03/2021	N
SO0049-018P	OB03IMS1	1.05	g		L	SL	IM	SF	02/03/2021	N
SO0049-018S	OB03IMS1	1.03	g		L	SL	IM	SF	02/03/2021	N
SO0049-019	OB03IMS1	1.22	g		L	SL	IM	SF	02/03/2021	F
SO0049-020	OB03IMS1	1.04	g		L	SL	IM	SF	02/03/2021	"
SO0049-021	OB03IMS1	1.07	g		L	SL	IM	SF	02/03/2021	"
SO0049-022	OB03IMS1	1.02	g		L	SL	IM	SF	02/03/2021	"
SO0049-023	OB03IMS1	1.08	g		L	SL	IM	SF	02/03/2021	"
SO0049-024	OB03IMS1	1.06	g		L	SL	IM	SF	02/03/2021	"
SO0049-025	OB03IMS1	1.00	g		L	SL	IM	SF	02/03/2021	"
SO0049-026	OB03IMS1	1.47	g		L	SL	IM	SF	02/03/2021	"
SO0049-027	OB03IMS1	1.21	g		L	SL	IM	SF	02/03/2021	"
SO0049-028	OB03IMS1	1.44	g		L	SL	IM	SF	02/03/2021	"
SO0049-029	OB03IMS1	1.06	g		L	SL	IM	SF	02/03/2021	"
SO0049-030	OB03IMS1	1.19	g		L	SL	IM	SF	02/03/2021	"
SO0049-031	OB03IMS1	1.14	g		L	SL	IM	SF	02/03/2021	"
SO0049-032	OB03IMS1	1.35	g		L	SL	IM	SF	02/03/2021	"
SO0095-003	OB03IMS1	1.05	g		L	SL	IM	SF	02/03/2021	"
SO0095-004	OB03IMS1	1.22	g		L	SL	IM	SF	02/03/2021	"
SO0095-006	OB03IMS1	1.19	g		L	SL	IM	SF	02/03/2021	"
SO0095-007	OB03IMS1	1.33	g		L	SL	IM	SF	02/03/2021	—

REVIEWED
EP 2/4/21
KATAHDIN ANALYTICAL
METALS SECTION

Client IDs verified -SF

QA-065-Revision 3 - 09/28/2018 Digestion performed by: SF/CD On: 2/3/21 Page: OB008 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

ATTACHMENT 1

MODIFICATIONS FOR 8330B PREPARATION & DIGESTION

4.0 APPARATUS AND MATERIALS – additional materials

4.1 Sieves - #10 mesh (2 mm) – 8" diameter with covers and collection trays.

4.2 Aluminum drying trays with drying rack

4.3 Heavy duty aluminum foil

4.4 Stainless steel scoopulas

4.5 Dust mask

9.0 PROCEDURES – additional procedures

Prior to the digestion of samples (section 7.1 in SOP):

Spread the entire aliquot of soil onto a drying tray lined with heavy duty aluminum foil and dry in air at room temperature or colder to a constant weight (last two successive dry weights within 3% RPD). Trays should be placed in rack for drying. Record all weights in the Sample Drying Logbook.

Note: Hydric soils and sediments with high moisture content may take several days to dry to constant weight.

Remove the oversize fraction by passing it through a 10-mesh (2 mm) sieve. Be sure to break up caked up soil with a gloved hand. Weigh both fractions – oversize and <2mm. Record all weights in the Sieving & Grinding Logbook.

To obtain a subsample, the entire sample must be mixed with a stainless steel scoopula and spread out on a clean surface (aluminum tray lined with foil) so that it is only 1 or 2 cm thick - preferably in a fume hood designed to prevent the spread of dust and possible inhalation or residue losses. Using the scoopula, obtain at least 30 different increments, i.e., portions (~0.3 g) from randomly chosen locations throughout the entire sample profile for a total of ~10 g. Mix this subsample one more time with the scapula and then obtain an aliquot for metals digestion (beginning with section 7.3).

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-608
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Bento Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hufstetler Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DH	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chkstd. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAVY award findings. Updated Sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up Updated tables to reflect changes in Standard Concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for Compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10
12	Revised Tables 4 → 8 with the following information: -Add palladium and gold; removed tungsten and uranium; removed Stock Standard ~~ICP-CICV~~ 200.7ICS-1; changed stock standard ICP-CICV-3 to CL-CAL-3. Added references to section 10.	LAD 04/22/11 LAD	09/11	09/11
13	The changes above had not been finalized in SOP-12. Sect. 9 - Added MDL, LOD and LOQ information. Added Attachment 2 - Analysis of Palladium by SW846 6010	LAD	04/12	04/12

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 9 & Table 1 - Fixed typos.	Jessie Dimond	05/13	05/13
15	Sect. 10 – Updated references. Added Table 3 – DoD QSM Ver. 5.0 QC requirements. Renumbered rest of Tables. Updated Tables 6-8. Changed KAS INC. to KAS LLC.	Jessie Dimond	12/14	12/14
16	Sect. 5 and 7 – Corrected Table references. Tables 5, 6, 7 & 8 – Updated Standards concentrations and sources.	Jessie Dimond	05/16	05/16
17	Sect. 1 and 6 - Added tissue matrix	Jessie Dimond	07/16	07/16
18	Sect. 8.1- changed reagent spiked water to calibration blank solution. Sect. 10- update method references.	Jessie Dimond	09/17	09/17
19	Updated Tables 6 & 7 with correct concentrations for some of the elements, Removed Table 2 – DoD QSM QC Requirements	Jessie Dimond	01/19	01/19
20	Sect. 7.8 – Added that all samples that exceed 90% of linear range must be diluted. Sect. 5.4 – Addition od Tritonx to calibration blank solution is used for instrument flush solution.	Jessie Dimond	04/20	04/20
21	Changed internal standard concentration from 5 mg/L to 0.50 mg/L in Section 5.8.	Jessie Dimond	01/21	01/21

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
22	Sect. 5 – Updated IS prep. Sect 7.5 – Updated frequency of ICSA/ICSB. Sect 8, Table 4, Table 6 & Table 9 – Added LRS 1 & 2. Table 9 – Added CCV & CCB	Lesie Dimond	09/22	09/22

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-22**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-22**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, LLC personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, biological tissue and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution

ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document

their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the maintenance logbook and run cover page and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual l and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by

choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates. Tritonx is added to calibration blank solution and is used to flush the system between standards and samples.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 4 and 5 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 0.50 mg/L yttrium internal standard solution – add ~~0.05~~ 2.0 mL 10000 mg/L yttrium stock standard to a 4L polystyrene container that is half filled with DI water. Add 200mL concentrated hydrochloric acid and 200mL concentrated nitric acid. Fill to 4L with DI water.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months
Tissue	P, G	25 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multi-element calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning of every run to verify the accuracy of the IEC factors. Refer to Section 8 and Tables 1 through 3 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Tables 1 through 3 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed. Tables 2 and 3 list the QC Check, minimum frequencies, acceptance criteria, corrective actions, flagging criteria and additional comments for work analyzed in accordance with DoD QSM versions 4.2 and 5.0.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of calibration blank solution, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis

process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within $\pm$ PQL if the PQL is greater than 0.01 mg/L, within $\pm$ 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

8.13 The upper limit of the linear dynamic range (LDR) must be analyzed daily using the linear range standards (LRS1 and LRS2) after each calibration anywhere during the analytical run. Refer to Section 8 and Tables 3 and 5 for additional information.

PREPARATION BATCH QC SAMPLES

8.14 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.15 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.16 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM 4.2 acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. If DoD QSM 5.0 acceptance criteria are being used, recovery for water and solid matrix samples must fall between the limits stated in Tables 3 & 4 of the QSM. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.17 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the

unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.
The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.17 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.18 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.18 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 6010C.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

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, 2016

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

List of Tables & Figures

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the LLCCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	
	Linear Dynamic Range verification (LRS1 and LRS2)	During each analytical run	Recovery within 10% of expected value	Do not report results greater than concentration of highest calibration standard

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike(MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-22	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 6)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	Calibration Standard 3	Claritas PPT	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	Inorganic Ventures	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn, Au	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 6)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 6)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 6)	1.0
Linear Range Standard 1 (LRS1)	1000 mg/L As, B, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn	High Purity Standards	2.0
	1000 mg/L Mo	High Purity Standards	0.5
	1000 mg/L Ag	High Purity Standards	0.2
	Concentrated HCl	J.T. Baker	2.0
Linear Range Standard 2 (LRS2)	10000 mg/L Al, Ca	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	3.0
	10000 mg/L Fe	High Purity Standards	2.5
	10000 mg/L Mg, Na	High Purity Standards	2.0
	10000 mg/L Si	High Purity Standards	0.5

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L Li, Sn, Au	High Purity Standards	1.0 each
	10000 mg/L K, Na	High Purity Standards	1.0 each
	1000 mg/l B	High Purity Standards	0.50
	1000 mg/l Zn	High Purity Standards	0.20
	1000 mg/L Cu	High Purity Standards	0.25
	10000 mg/L Si	High Purity Standards	0.20
	1000 mg/L Ti, Tl	High Purity Standards	0.15 each
	1000 mg/L Se, Mo, Co, Ni, Ag, Sr, V, Cr	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba, Be, Cd, Mn, Pb	High Purity Standards	0.05 each
	10000 mg/L Fe, Ca, Mg	High Purity Standards	0.1 each
ICSAB-INT	1000 mg/L K,Na	High Purity Standards	4.0 each
	1000 mg/L B, Li, Mo,Sr,Sn,Ti, Au	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.40
ICP-INT STD (Intermediate)	10000 mg/L Si	High Purity Standards	2.5
	10000 mg/L Ca, Mg, Fe, Al, Na	High Purity Standards	2.4
	10000 mg/L K	High Purity Standards	1.5
	1000 mg/L Au, Li, Sn. Sr	High Purity Standards	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L									
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**LRS1**	**LRS2**
Aluminum	25	10	0.3	500	500	12.5	500			500
Antimony	1	0.4	0.008		0.6	0.5			20	
Arsenic	1	0.4	0.008		0.1	0.5			20	
Barium	1	0.4	0.005		0.5	0.5			20	
Beryllium	1	0.4	0.005		0.5	0.5			20	
Boron	1	0.4	0.05		0.5	0.5			20	
Cadmium	1	0.4	0.005		1.0	0.5			20	
Calcium	25	10	0.10	500	500	12.5				500
Chromium	1	0.4	0.01		0.5	0.5			20	
Cobalt	1	0.4	0.01		0.5	0.5			20	
Copper	1	0.4	0.025		0.5	0.5			20	
Iron	25	10	0.1	200	200	12.5		200		
Lead	1	0.4	0.005		0.05	0.5				250
Lithium	1	0.4	0.1		0.5	0.5			20	
Magnesium	25	10	0.10	500	500	12.5			20	
Manganese	1	0.4	0.005		0.5	0.5				200
Molybdenum	1	0.4	0.01		0.5	0.5			20	
Nickel	1	0.4	0.01		1.0	0.5			5	
Potassium	25	13.6	1		20	12.5			20	
Selenium	1	0.4	0.01		0.05	0.5				300
Silicon	25.5	10.0	0.2		2	12.75			20	
Silver	1	0.4	0.01		0.2	0.5				50
Sodium	25	10	1		20	12.5			2	
Strontium	1	0.4	0.01		0.5	0.5				200
Thallium	1	0.4	0.015		0.1	0.5			20	
Tin	1	0.4	0.1		0.5	0.5			20	
Titanium	1	0.4	0.015		0.5	0.5			20	
Vanadium	1	0.4	0.01		0.5	0.5			20	
Zinc	1	0.4	0.02		1.0	0.5			20	
Gold	1	0.4	0.1		0.5	0.5			20	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP Intermed STD	PQL-INT	ICSAB-INT
Aluminum	240	30	
Antimony		0.8	
Arsenic		0.8	
Barium		0.5	
Beryllium		0.5	
Boron		5	10
Cadmium		0.5	
Calcium	240	10	
Chromium		1.0	
Cobalt		1.0	
Copper		2.5	
Iron	240	10	
Lead		0.5	
Lithium	10	10	10
Magnesium	240	10	
Manganese		0.5	
Molybdenum		1.0	10
Nickel		1.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.0	
Sodium	240	100	400
Strontium	10	1.0	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	10
Vanadium		1.0	
Zinc		2.0	
Gold	10	10	10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	IV-28	QCS-26	CLPP-ICS-A	CLPP-ICS-B4	CL-CAL-3
Aluminum	100	100	5000		
Antimony	100	100		60	
Arsenic	100	100		10	
Barium	100	100		50	
Beryllium	100	100		50	
Boron	100	100			
Cadmium	100	100		100	
Calcium	100	100	5000		1000
Chromium	100	100		50	
Cobalt	100	100		50	
Copper	100	100		50	
Iron	100	100	2000		1000
Lead	100	100		5	
Lithium	100				
Magnesium	100	100	5000		1000
Manganese	100	100		50	
Molybdenum	100	100			
Nickel	100	100		100	
Potassium	1000	1000			1000
Selenium	100	100		5	
Silicon	50	50			
Silver	100	100		20	
Sodium	100	100			1000
Strontium	100				
Thallium	100	100		10	
Tin					
Titanium	100	100			
Vanadium	100	100		50	
Zinc	100	100		100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10	LRS1 (Linear Dynamic Range Verification)	Verify upper linear dynamic range limit
11	LRS2 (Linear Dynamic Range Verification)	Verify upper linear dynamic range limit
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-24	Analyze up to 10 samples	
25	CCV (Continuing Calibration Verification)	Check calibration stability
26	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 2

ANALYSIS OF PALLADIUM BY SW846 6010

Palladium may be analyzed by EPA Method SW846 6010C following the method outlined in this SOP. However, due to significant spectral interferences caused by addition of palladium to the calibration and check standards used in this method, palladium is added to aliquots of the regular standards as needed for analysis. Two stock standards (1000 mg/L) are currently kept for palladium analysis. One is purchased from High Purity Standards and is used for calibration, PQL, ICSAB, and CCV. The other is purchased from Inorganic Ventures and is used as the independent check standard (ICV). Analysts should add palladium stock to the regular standards according to the table below:

Name of Working Standard	Volume of Standard Aliquot (mL)	Volume of Palladium Stock Added (mL)	Concentration of Palladium (mg/L)	Source of Palladium Stock
Calibration Std.	50	0.05	1.0	High Purity
ICV	50	0.02	0.4	Inorganic Ventures
PQL	50	0.005	0.1	High Purity
ICSAB	50	0.025	0.5	High Purity
CCV	50	0.025	0.5	High Purity

Prior to starting the run, a palladium-only standard should be analyzed along with the iron and aluminum standards to evaluate interelement correction factors as outlined in Katahdin SOP CA-632, Section 7.1.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Jeb C. Bustin Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Murphy Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	DM	1.29.01	1/29/01
03 7471A	Changed Leeman PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised Sect. 10 to show correct reference material. Removed fig. 2. Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (CCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition, Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DM	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD OSM Version 4.1 QC Requirements	LAD	04/10	04/10
08	Sect. 4.6 - Changed thermometer type. Added LCSO - A LCS prepped using aqueous mercury LCS spike. Updated type of marker used to label digestion bottles. Updated corrective action for failing PQL standard.	LAD	12/10	12/10
09	Sect. 7 - Changed calibration digestion from digestion of all points to digestion of high point and dilutions rest. Changed prep from 3 to 0.2 g aliquots to 1 ~ 0.6 g aliquot. Added additional prep info. Added Serial dilution and PDS to sect. 8, Added MDL, LOD, LOQ info. to Sect 9. Updated and added references to Sect. 10.	LAD	04/12	04/12
10	Sect. 7 - Corrected Calibration preparation, changed digestion temperature to 95 +/- 3°C. Sect. 10 - Added and updated references. Added Table 3 - DoD OSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 4 - Added Snap-top containers and digestion tubes. Sect 5 - Updated Aqua Regia prep. Sect. 7 - Added heat block and digestion tube instructions, minor edits. Removed Table 2, updated fig. 1, added fig. 4 Changed ~~KASINC~~ KAS throughout. KASINC to LAD 10/25/17	LAD	10/17	10/17
12	Sect. 7 - Updated to reflect current Calibration and Independent Calibration verification Standard preparation. Corrected typos	LAD	01/19	01/19
13	Sect. 7 - ~~Corrected~~ Corrected Calibration high Standard and ICV preparation. Sect 8 - Added if CCV or LCS fail high and samples ND - report and narrate	LAD	10/19	10/19
14	Changed water bath to Hotblock throughout SOP	LAD	04/20	04/20

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
15	Sect. 4.3 – Added make and model of the balance.	Jessie Dimond	06/20	06/20
16	Minor changes to equipment descriptions in Sections 4.1 and 4.4. New bench sheet in Figure 1.	Jessie Dimond	02/21	02/21
17	Sect 4 - Updated for new mercury instrument. Sect 4, 7, Table 1 and 2 –Removed information that is in the Instrument operation and maintenance SOP. Removed routine analysis of a SRM sample. Removed Standard Additions	Jessie Dimond	09/22	09/22

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-17, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-17, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

.

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis of solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, sludges and tissue under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process. LCSS utilizes the standard reference material. LCSO is spiked with aqueous mercury LCS spike.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps or polyethylene digestion tubes, for use as digestion vessels.

4.2 Hotblock unit capable of maintaining a constant temperature of 95 ±3°C.

4.3 Analytical balance capable of weighing to 0.01 g(Ohaus, model SPE402 or equivalent).

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 1 to 10 uL, 10 to 100 uL, 100 to 1000 uL, 1000 uL to 5000 uL. Calibrated Eppendorf of Fisher Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Thermometer, NIST-traceable, covering the range from -10^{o} to 110^{o} C, for monitoring the temperature of the Hotblock. Mercury-filled thermometers are not acceptable for use in the metal's laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 Teledyne M-7600 Mercury analyzer and associated peripherals and parts.

4.9 4oz graduated snap-cap container, 120 mL capacity.

Refer to Katahdin SOP CA-640, current revision, "Operation and Maintenance of the Teledyne M-7600 Mercury analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 1:1 Aqua regia: Prepare an appropriate amount immediately before use. Start with 4 parts laboratory reagent grade water, carefully add one part of concentrated HNO_3 and then three parts of concentrated HCl in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood. Record preparation in "Metals Preparation Laboratory - Reagent Preparation Logbook". Refer to Figure 3 for a cop of a page from this logbook

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 7% Hydrochloric acid. This intermediate standard is used to prepare calibration standards, matrix spikes,

CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook. Intermediate Mercury Standard A must be prepared monthly and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 7% Hydrochloric Acid The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook Intermediate Mercury Standard B must be prepared monthly and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 2 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights

and final volumes, pertinent observations, etc.) must be recorded on this bench sheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.2 Using an industrial marker with super permanent ink, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control sample and matrix spike sample to be digested.

7.3 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.4 Prepare an appropriate number of laboratory control samples (CSO) by weighing appropriate by adding 500 uL of Intermediate Mercury Standard A to 1.0 g Teflon chips into a labeled digestion bottle. The mercury concentration of the LCSO will be 5.0 ug/L.

7.5 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.6 Preparation blanks, laboratory control spike and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP. Calibration standards are not digested.

SAMPLE PREPARATION AND DIGESTION

7.7 Do not decant any water on the sediment sample. **Note:** Some workorders may have to decant samples in the work notes. This is **always** done during login and **never** at the time of extraction. Samples decanted during login will be marked accordingly.

Mix sample with a wooden spatula to ensure homogeneity of the sample. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“ for more detailed guidance on sub-sampling to ensure reproducibility.

Weigh an approximate 0.6 0.8 g portion of untreated, homogenized sample from the sample container and place in the bottom of a labeled digestion bottle.

7.8 Add 10 mL of 1:1 Aqua Regia to each sample, standard, and QC sample. Place bottles in a heat source located in a fume hood and heat for 2 minutes at 95 ±3°C. Remove the bottles from the Hotblock and allow them to cool in a fume hood for 8 minutes.

7.9 For glass mercury bottle preparation, add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For mercury digestion tube preparation, add 20 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. For Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the Hotblock and heat for 30 minutes at 95°C. Record initial and final time and temperatures on the mercury preparation bench sheet.

7.10 Remove the bottles from Hotblock and allow them to cool in a fume hood. If any of the samples have become colorless during heating, reprep using a lower initial mass.

7.11 For glass mercury bottle preparation, add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

7.12 For mercury digestion tube preparation, transfer sample into 4oz snap cap. Add 6ml sodium chloride-hydroxylamine hydrochloride solution into each digestion vessel, swirl to mix with remains of sample, and pour into snap cap with corresponding sample. Swirl snap cap to mix.. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Bring sample to 100 mL final volume with reagent grade water. Wait at least 30 seconds before proceeding with analysis.

DATA REDUCTION AND REPORTING

7.13 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported – the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{I \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.14 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate

analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

PREPARATION BATCH QC SAMPLES

8.4 Preparation blank (PBW or PBS), consisting of Teflon boiling chips carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.5 A laboratory control sample (CSO), consisting of Teflon boiling chips spiked with 500 uL of Intermediate Standard A carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples that are supplied by the vendor of the solid reference material. The results of the LCSO must fall within 80% - 120% of its true value which is 5.0 ug/L. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS or LCSO must be within 80% - 120%.

Samples that are below the reporting limit may be reported if the LCSS or LCSO reads greater than 120%.

SAMPLE MATRIX QC SAMPLES

8.6 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.7 Serial Dilution – A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L - S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.8 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

8.9 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantiaion (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

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, 2016.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration > PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS or LCSO)	One per digestion batch of 20 or fewer samples.	LCSS: Recovery within vendor-supplied acceptance limits. LCSO: Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	1)Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes or duplicate samples.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-17	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

SF 2/8/21

Reagents and Consummables Information: **Method:** 7471

Aqua Regia: MR29604 Digestion Vessels: MLDOP260000046

KMNO4: MR9955 NH2OH-HCl: MR22948 Boiling Stones: MSR207

Standards/Spiking Information:

1ppm A : MW19978
Pipet 1ppm B : MW19975
LCSO = 500uL of 1ppm A to 100mL
Spike(S/P) = 100uL of 1ppm A to 100mL
ICV = 600uL of 1ppm B to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL
Balance ID: 09 LCSS: —

Heat Source ID : Loaner
Start Time: 1200 /Temp. 94.4 °C
End Time: 1230 /Temp. 92.8 °C
Thermometer ID/Pos: DI676 / 5:3

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSOOB05HGS1	OB05HGS1	0.6	g	0.1	L	SL	HG	CD	02/05/2021	—
PBSOB05HGS1	OB05HGS1	↓	g	↓	L	SL	HG	CD	02/05/2021	—
SO0506-001	OB05HGS1	0.77	g	↓	L	SL	HG	CD	02/05/2021	B
SO0594-001	OB05HGS1	0.77	g	↓	L	SL	HG	CD	02/05/2021	E VA
SO0597-001	OB05HGS1	0.69	g	↓	L	SL	HG	CD	02/05/2021	F
SO0597-002	OB05HGS1	0.81	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-003	OB05HGS1	0.74	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-004	OB05HGS1	0.77	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-005	OB05HGS1	0.67	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-006	OB05HGS1	0.80	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-007	OB05HGS1	0.64	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0597-008	OB05HGS1	0.73	g	↓	L	SL	HG	CD	02/05/2021	↓
SO0650-002	OB05HGS1	0.71	g	↓	L	SL	HG	CD	02/05/2021	B
SO0659-001	OB05HGS1	0.60	g	↓	L	SL	HG	CD	02/05/2021	—
SO0597-001P	↓	0.68								F
SO0597-001S		0.71								F

Client ID's verified CD 2-5-21

REVIEWED
EP 2/8/21
KATAHDIN ANALYTICAL
METALS SECTION

QA-067-Revision 3 - 09/28/2018 Digestion performed by: CD On: 2-5-21 Page: OB021 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 2

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

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FIGURE 3

EXAMPLE REAGENT PREP LOGBOOK PAGE

REAGENT PREP LOG - METALS PREP LAB

Reagent ID	Reagent Name	Prep Date	Exp. Date	Prep Initials	Component	Lot Number	Amount Added	Prep Notes
MR1863	$SnCl_2$ - HCL	9-8-17	12-8-17	AMJ	$SnCl_2$	MSR66/MSR76		FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc HCL	MSR68	280mL	↓
MR1864	1:1 Aqua ~~Regia~~ Regia	9-12-17	9-13-17	AMJ	Conc. HCL	MSR68	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR75	50mL	↓
MR1865	NH_2OH - HCL (12%w/v)	9-14-17	AMJ 9/14/17 9-14-17	AMJ	NH_2OH	MSR57/MSR58	480.01	FV= 4000mL w/ Reag H_2O
↓	↓	↓	↓	↓	NaCL	MSR26	480.01	↓
MR1866	1:1 aqua Regia	9-18-17	9-19-17	AMJ	Conc. HCL	MSR68	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR77	50mL	↓
MR1867	5% HNO_3	9-18-17	9-18-18	AMJ	Conc. HNO_3	MSR77	500mL	FV= 10L w/ Reag H_2O
MR1868	5% (M/v) $KMNO_4$	9-18-17	AMJ 9/18/18 9-12-18	AMJ	$KMNO_4$	35309	200.00g	FV= ~~500~~ 4L w/ Reag H_2O AMJ 9/18/17
MR1869	1:1 aqua Regia	9-20-17	9-21-17	AMJ	Conc HCL	MSR73	150mL	FV= 400mL w/ Reag H_2O
↓	↓	↓	↓	↓	Conc. HNO_3	MSR77	50mL	↓

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ADDENDUM A

TISSUE PREPARATION

Prepare tissue samples in the same manner as soils with the following exceptions.

Tissue Preparation	EPA 7471B
Weigh 0.2 to 0.3 g portions of each sample	Weigh 0.5 to 0.6 g portions of each sample
Add 4 mL on concentrated h2so4 and 1 mL of concentrated HNO3 to each bottle	Add 5 mL H2O and 5 mL of Aqua Regia
Heat to 58°C for 30-60 minutes	Heat at 95°C for 2 minutes
Cool to 4°C in an ice bath	Cool to room temperature
Add 5 mL of potassium permanganate solution in 1 mL increments	Add 50 mL H2O and 15 mL potassium permanganate
Add an additional 10 mL potassium permanganate or more to maintain oxidizing conditions	
Add 8 mL of potassium persulfate	
Allow to stand overnight at room temperature	Heat at 95°C for 30 minutes

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hoffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02·16·05	02·16·05
02	Updated Fig. 1 - new prep Logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

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Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoDQSM version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.6 - Changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard correction action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12
08	DoDQSM 5.0 References added. Sect. 7.4 and Table 3 - Updated Calibration Standard prep - removing digesting all Standards. Added to digest high point	LAD	06/14	06/14
09	Updated figure 1. Change title of section 5.0. Update method references for NELAC and DoD. Minor additions to sections 4.1, 4.2, 4.6, 7.1, 7.10, 7.12.	LAD	09/17	09/17
10	Removed DoDQSM 4.2 QC Requirement Table. Added DoDQSM 5.0/5.1 QC Requirement Table. Updated references. Updated Logbook Example	LAD	11/18	11/18
11	Sect. 7 - Updated to reflect current Calibration and Independent Calibration Verification Standards	LAD	01/19	01/19
12	Removed use of Pyrex media bottles and VOA vials. Changed water bath to heating Block. Added the addition of 6 ml sodium chloride-hydroxylamine hydrochloride solution to standards. Table 1 - Added Serial dilution and post digestion spike.	LAD	06/20	06/20
13	Changed instructions for making 5.0 mg/L Calibration Std., in Sect 7.4, to double the final volume.	LAD	02/21	02/21

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Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect 4 - Updated for new mercury instrument. Sect 4, 7, Table 1 and 2 –Removed information that is in the Instrument operation and maintenance SOP. Removed routine analysis of a SRM sample. Removed Standard Additions	Leslie Dimond	09/22	09/22

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-14,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-14,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services personnel for the digestion and analysis of aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer.

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3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL, 50 mL or 70 mL digestion tubes and appropriate watch glasses, for use as digestion vessels.

4.2 Heating block capable of maintaining a constant temperature of 95^{o} C.

4.3 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL, 1mL to 5mL, and 2mL to 10mL calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.4 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.5 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50^{o} to 750^{o} C, for monitoring the temperature of the Heat Block. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.6 50 mL digestion tubes appropriate for the heat block

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4.7 Teledyne M-7600 automated mercury analyzer and associated peripherals and parts

4.8 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-640, current revision, "Operation and Maintenance of the Teledyne M-7600 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS AND STANDARDS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: In a 2L disposable container dissolve 100 g of potassium permanganate in 2 L laboratory grade reagent water. The container is then disposed of "L" waste.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 100g of potassium persulfate in 2L laboratory grade reagent water.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 240 g sodium chloride and 240 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 2 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. ~~Reagent should be labeled as suitable for use in mercury determination.~~

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 7% Hydrochloric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

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5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 7% Hydrochloric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 240mL or 300mL graduated snap caps. These are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 50 mL digestion tubes.

The disposable graduated 50mL centrifuge tubes in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation

date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this Bench sheet, Which will be reviewed, scanned, and saved to the appropriate folder prior to reporting. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Prepare one prep blank(PBW) for every 20 sampleS by adding 25 mL of laboratory grade reagent water to labeled vials.

7.4 Prepare one laboratory control sample (LCSW) for every 20 samples by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L. If no matrix QC is requested for batch than and laboratory control sample duplicate (LC2W) may be prepped instead and is prepared the same as the LCSW

7.5 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.6 Preparation blanks, laboratory control spikes and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the~~ media bottles~~ Large snap caps are four times those listed in sections 7.10 through 7.13 but the standards are not heated.

SAMPLE PREPARATION AND DIGESTION

7.7 Using a graduated disposable dosecup or pour directly into graduated digestion tube, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, ~~swirling to mix after each addition.~~ Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes.

7.8 Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

Some samples may require dilution to 25 mL with potassium permanganate for digestion to be performed in the digestion vessel. Prepare method blank and LCS

with equal amounts of potassium permanganate to check for potential mercury contamination.

7.9 Add 2 mL of potassium persulfate solution to each sample, add ribbed watch glasses, and place them in a preheated heating block. Monitor the temperature of the ~~bath~~ Block with a thermometer throughout the digestion. The temperature of the water bath will fall below 90-95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 90-95^{o} C, continue heating the samples at 90-95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.10 Remove bottles from the heating black and allow to cool for at least 8 minutes. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 90-95^{o} C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.11 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and cap and shake until color change is complete, then loosen cap to pressure buildup. This will reduce the excess permanganate, and the sample will change from purple to colorless. After color change, samples are filled to 40 mL with laboratory grade reagent water and mixed well. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.12 Digested mercury samples are analyzed using the Teledyne M-7600 Automated Mercury Analyzer. Analysis is automated and is controlled by the Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-640, “Operation and Maintenance of the Teledyne M-7600 Automated Mercury Analyzer”.

DATA REDUCTION AND REPORTING

7.13 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.14 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.15 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a “U” qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific

product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be prepared each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110%

of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within

80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, current Version.

The 2016 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Serial Dilution	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.
	Post-Digestion Spike	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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Table 2

DOD QSM REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank. CVAA/Mercury: minimum 5 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-14	USEPA METHOD 7470
Reagents	1) Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	1) Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	2) Sampling and gas stream switching performed automatically by mercury analyzer. 3) Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	2) Sampling and gas stream switching performed manually by analyst. 3) Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	4) Known reference sample (ICV) analyzed daily. 5) Calibration verified after every 10 samples with CCV.	4) Known reference sample analyzed quarterly. 5) Calibration verified after every 20 samples.
QC - Calibration Blanks	6) Acceptance criteria employed for 245.1: $\pm$ PQL	6) Acceptance criteria stated in 245.1: $\pm$ MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagents and Consumables Information: **Method:** 7470

HNO3: MSR149 H2SO4: MSR103 Digestion Vessles: 180905

KMNO4: MR2174 K2S2O8: MR2142 NH2OH-HCl: MR2168

REVIEWED JS 11.13.18 KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:

1ppm A : MW18167

Pipet 1ppm B : MW18158

MH LCSW = 125uL of 1ppm A to 25mL

MH Spike(S/P) = 25uL of 1ppm A to 25mL

MB ICV = 600uL of 1ppm B to 100 mL

MB S10.0 = 1000uL of 1ppm A to 100 mL

Heat Source ID : B

Start Time: 1142 /Temp. 92 °C

End Time: 1542 /Temp. 90 °C

Thermometer ID/Pos: ALL25, 24

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Bottle
LCSWLK12HGW1	LK12HGW1	0.025	L	0.025	L	AQ	HG	AMJ	11/12/2018	
PBWLK12HGW1	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0847-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-006P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-006S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	S
TL0847-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-009	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-010	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-011	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-012	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0847-013	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	G
TL0912-001	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001P	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-001S	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	C
TL0912-002	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	A
TL0912-003	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-004	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-005	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-006	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-007	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	
TL0912-008	LK12HGW1		L		L	AQ	HG	AMJ	11/12/2018	

JS 11.13.18

QA-066-Revision 4 - 09/28/2018 Digestion performed by: AMJ On: 11/12/18 Page: LK061 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Figure 2

Digestion Procedure Overview

1. Fill digestion tubes with 25 mL sample or DI and respective spikes for QC

PBW- 25mL DI

LCSW- 25mL DI + 125uL HgA

S, P- 25mL native sample + 25uL HgA

2. Add 0.625mL HNO_3 and 1.25mL H_2SO_4

3.Add 3.75mL $KMnO_4$ and allow to sit for 15 mins-

-If purple/ metallic color persists, proceed to step 4

-If sample turns brown add another 3.75mL addition to sample and BQC

4. Add 2mL K_2SO_8

5. Cover with ribbed watch glasses and heat at 90-95 degrees for 2 hours

6. Remove from heat block and cool for 8 min.

-If any sample(s) contains no excess $KMnO_4$ (no brown color); add another 3.75mL of $KMnO_4$ to sample(s) and BQC and heat for another 2 hours.

7. Add 1.5 mL NaCl- NH_2OH; cap and mix until $KMnO_4$ is gone

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Prepared By: George Brewer Date: 03/14/05

Approved By:

Department Manager: George Brewer Date: 03/14/05

Operations Manager: Deborah J. Nadeau Date: 3.14.05

QA Officer: Leslie D'Amond Date: 031405

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added expirations dates (1yr) for SPLP fluid s Added DOC requirements Revised TCLP/SPLP logbooks - making it usable for both TCLP/SPLP and record pH, expiration dates	LAD	01/07	01/07
02	Updated figures 6 → 8 with current logbook pages.	LAD	04/08	04/08
03	Updated or added references to sections 1.3, 9 and 10. Updated logbook page.	LAD	06/10	06/10
04	Figure 8 - Updated with new logbook. Revised text references to logbook throughout. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added, removed and edited references	LAD	04/12	04/12
05	Changed KAS INC. to KAS throughout. Updated Figures 6 & 7. Updated DoD references	LAD	03/15	03/15

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sec. 4 – added thermometer. Sec. 5.4 – changed HCl to HNO_3. Moved table from Sec 6.4 to end (Table 4). Updated logbook pages. Changed wording in sections 6, 7 and 8 for clarity and to match CA-510. Updated SPLP matrix spiking (per PBT).	Lesie Dimond	03/18	03/18
07	Minor edits to fix typographical errors, updated references and logbook example	Lesie Dimond	01/20	01/20

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-620-07**, titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-620-07** titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes.**

Recipient: _____ Date: _____

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services, personnel for SPLP extraction of samples for inorganic and non-volatile organic components using US EPA Method 1312 (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The SPLP (Synthetic Precipitation Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in SPLP extractions. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in SPLP extractions to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be aware of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method may not be precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's 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 Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the

laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal and Pollution Control

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes from SPLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Plan and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the initial liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. If the sample is a soil, the composition of the extraction fluid employed depends on the region of the country where the sample site is located. If the sample is a waste or a wastewater, the extraction fluid used is a pH 4.2 solution. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of SPLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the SPLP procedure and are discussed in detail in Section 8.0: Quality Control and Acceptance Criteria.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 revolutions per minute (rpm) – see Figure 1. The RPM of each extractor must be manually verified by counting the number of rotations for 1 minute. The rotation rate of each extractor is verified before each use, and the results are recorded in the Non-Volatile TCLP/SPLP Extraction Logbook (Figure 7). If the measured rotation rate of an extractor is outside the range 30 ± 2 rpm, it must be taken out of service until it can be repaired.

4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated plastic (e.g. Teflon) if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.

4.3 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 µm, 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 1N HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.

4.5 pH meter accurate to ± 0.05 units at 25 °C. The pH meter must be calibrated on each day of use.

4.6 Laboratory balance accurate to within ± 0.01 g

4.7 Beakers, glass, 500 mL

4.8 Watch glasses, appropriate diameter to cover beakers

4.9 Magnetic stirrer

4.10 Room thermometer capable of recording min/max temperatures over a 24 hour cycle

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5.0 REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Laboratory reagent grade water - Reagent water should be monitored periodically for impurities.

5.2 Sulfuric acid, concentrated (H_2SO_4) – reagent grade

5.3 Nitric acid, concentrated (HNO_3) – reagent grade

5.4 Nitric acid, 1N, for acid-washing filters. Dilute 63 mL reagent grade HNO_3 to 1000 mL with laboratory reagent grade water.

5.5 Sulfuric acid (0.6 weight percent) / nitric acid (0.4 weight percent) mixture – Add 0.60 ± 0.05 g concentrated H_2SO_4 and 0.40 ± 0.05 g HNO_3 to a 100 mL volumetric flask half-filled with laboratory reagent grade water. Swirl to mix and bring to a final volume of 100 mL with laboratory reagent grade water.

5.6 SPLP Fluid #1 – Add approximately 15 L of laboratory reagent grade water to a clean graduated 20 L polyethylene carboy reserved for this fluid. Add approximately 5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 20 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 4.20 ± 0.05. If necessary, add more acid solution to lower the pH, or remove some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #1 is used to determine the leachability of soils from sites east of the Mississippi River, and the leachability of wastes and wastewaters. The fluid may be used for up to one year from the preparation date.

5.7 SPLP Fluid #2 - Add approximately 4 L of laboratory reagent grade water to a clean graduated 5 L polyethylene carboy reserved for this fluid. Add approximately 1.5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 5 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 5.00 ± 0.05. If necessary, add more acid solution to lower the pH, or dump out some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #2 is used to determine the leachability of soils

from sites west of the Mississippi River. The fluid may be used for up to one year from the preparation date.

5.8 SPLP Fluid #3 – This fluid is laboratory reagent grade water (Section 5.1) and is used to determine cyanide leachability.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile TCLP/SPLP Extraction Logbook (Figure 7) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the SPLP Fluid Preparation and Use Logbook (Figure 6). Upon preparation, each new batch of extraction fluid is assigned a batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the SPLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4 °C and opened immediately prior to SPLP extraction.

6.3 SPLP extracts should be prepared for analyses and analyzed as soon as possible following SPLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

6.4 See Table 4 for sample holding times for non-volatile SPLP extraction and analysis.

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 2 and 3. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This

aliquot may not actually undergo SPLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the SPLP extraction, Section 7.4.

All information and measurements pertaining to SPLP extractions are recorded in the Non-Volatile SPLP Extraction Logbook (Figure 7). In the following procedure, the section or column of the Non-Volatile SPLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Column C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section II**) - Percent solids is defined for SPLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.

If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Column A**) and the container that will receive the filtrate (filtrate vessel) (**Column B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.2 Weigh out a subsample of the waste (100 g minimum) and record the combined weight of the weigh boat and waste (**Column C**).

7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

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7.1.6 Weigh the weigh boat and any residue clinging to it (**Column D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column E**).

7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.1.8 Weigh the filtrate vessel and its contents (**Column F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column G**).

7.1.9 Calculate the percent wet solids as follows (**Column H**):

$$\text{Percent wet solids} = \frac{\text{(Total weight of waste)-(Weight of liquid phase)}}{\text{Total weight of waste}} * 100$$

7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighings yield the same value within ± 1%. Record the weight of the filter and dry solids (**Column I**).

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NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Column J**).

7.2.4 Calculate the percent dry solids as follows (**Column K**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile SPLP Extraction Logbook (e.g. "No apparent solids present – dry solid weight is due to entrained non-volatile liquid"), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm^3. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a surface area or particle size as described above. Perform particle size reduction on the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 For soils, if the sample is from a site that is east of the Mississippi River, SPLP Fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, SPLP Fluid #2 should be used.

7.4.2 For wastes and wastewaters, SPLP Fluid #1 should be used.

7.4.3 For cyanide containing wastes or soils, SPLP Fluid #3 must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

SPLP EXTRACTION FOR NON-VOLATILES

7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of SPLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e. is 100% solid), weigh out a subsample of the waste (100 gram minimum), record the weight (**Section II**), and proceed to Section 7.5.11. If the sample is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Column L**).

7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.

7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Column M**). If the waste contains < 0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the SPLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing > 0.5% dry solids, information is obtained in Section 7.1 (Percent Solids Determination) to calculate the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the SPLP extract.

7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.

7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.5.7 Weigh the weigh boat and any residue clinging to it (**Column N**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Column O**).

7.5.8. Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through he filter in any 2-minute interval, slowly increase in pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.5.9 Weigh the filtrate vessel and its contents (**Column P**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Column Q**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Column R**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Column S**).

7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in

Section 7.3. Describe the particle size reduction process in **Section IV** of the logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

Weight of extraction fluid = (20) (Weight of wet solids)

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID and the pH (measured on day of use) in the Comments section **(Section V)** of the logbook. Record the amount used in **Section II** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 RPM during the extraction period of 18 ± 2 hours at 23 ± 2 °C. Record the extraction start and end times and the room temperatures (min/max throughout extraction process) in **Section I** of the logbook. In order to maintain the required temperature range throughout the extraction, a temperature controlled extraction case is used that has temperature maintained by an individual heating and cooling system.

NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Sections 7.5.6 and 7.5.8. For final filtration of the SPLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

7.5.14 Prepare the SPLP extract as follows:

7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.3 If the initial liquid phase of the waste, as obtained prior to extraction from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in **Section V** of the logbook. Individually analyze these two liquids, collectively defined as the SPLP extract, and combine the results mathematically, as described in Section 7.6.

7.5.15 Following collection of the SPLP extract, the pH of the extract should be measured and recorded (**Section II**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. All other aliquots must be stored under refrigeration (4 °C) until analyzed.

7.6 The SPLP extract shall be prepared and analyzed according to appropriate analytical methods. SPLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.6 Compare the analyte concentrations in the SPLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

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USEPA Method 1312 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel.

8.1.1 After SPLP extraction, SPLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Table 4). For this reason, it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for SPLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 1391. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full SPLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 1391. Because the holding time for the previous SPLP method blank for pesticides and herbicides has expired, a new SPLP method blank must be

extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 1391 has already been analyzed for these constituents.

8.1.2 Each SPLP method blank is identified in the SPLP extraction logbooks by code. The first three characters are "PBT", which stands for "Preparation Blank – TCLP/SPLP". Following this is the preparation number of the extraction fluid (e.g., 1391), which is unique to the extraction date for a particular batch of fluid. The last character is a letter, starting with "A" and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, "PBT1391A" refers to the first SPLP method blank extracted using fluid from Batch 1391; "PBT1391B" refers to the second SPLP method blank extracted using the same fluid. The extraction date of each SPLP method blank is recorded in the Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook. For every SPLP method blank prepared, at least one matrix-spiked aliquot must be prepared with a sample associated with that SPLP method blank. Record the sample chosen and spiking amounts in the logbook. See Section 8.2 for matrix spiking procedure.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. EPA Method 1312 requires one MS/MSD per analytical batch. Because the laboratory charges for the preparation and analysis of SPLP matrix spikes, selection of samples for SPLP matrix spiking is left to the discretion of the client. Follow the matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 3 and Figures 4 and 5.

8.2.1 Matrix spikes are to be added after filtration of the SPLP extract and before any preservation. Matrix spikes should not be added prior to SPLP extraction of the sample.

8.2.2 Instructions for preparing SPLP matrix spikes for metals analysis are contained in Table 3. Instructions for preparing SPLP matrix spikes for organics analyses are contained in Figures 4 and 5. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of SPLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\text{Recovery (\%)} = 100\ (X_s - X_u)/K$$

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where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the SPLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the SPLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall be followed.

9.0 METHOD PERFORMANCE

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 level, concentration, or quantity of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 1312 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Third Edition, Final Update I (7/92), Method 1312

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, 10/06/2010.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

List of Tables and Figures

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Synthetic Precipitation Leaching Procedure (SPLP) / EPA 1312	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid. One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods. Refer to individual analytical methods	Prepare fresh extraction fluid and repeat SPLP extraction of all associated samples. Remove extraction vessel from service.
	Matrix Spike	One per analytical batch (required by method 1312). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within ± 20% of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-620-07	EPA METHOD 1312
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike **recommended** for each waste type and analytical batch.	Matrix spike **required** for each waste type and analytical batch.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

SPLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
SPLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.20
	CLPP-SPK-INT1	Lab Prepared (see below)	2.0
SPLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.20

Note: Spiking must be performed after SPLP extraction and before preservation.

PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	**CONCENTRATION IN SOLUTION, mg/L**			
	SPLP Matrix Spike	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**1000 ug/L Hg Std.**
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 4

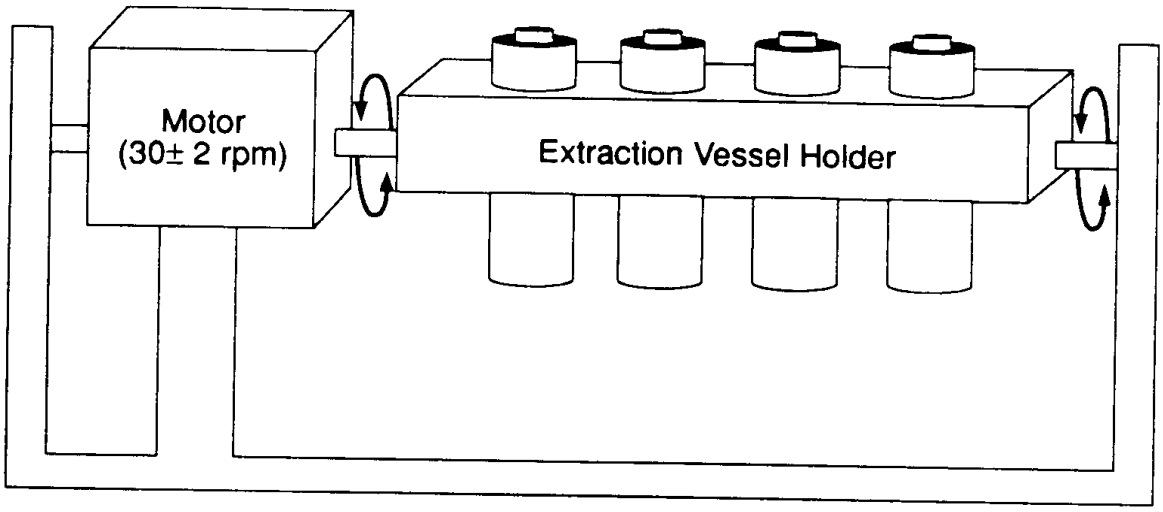
SPLP HOLDING TIMES SUMMARY

SPLP PARAMETER	FROM COLLECTION TO SPLP EXTRACTION	FROM SPLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 1

ROTARY AGITATION APPARATUS



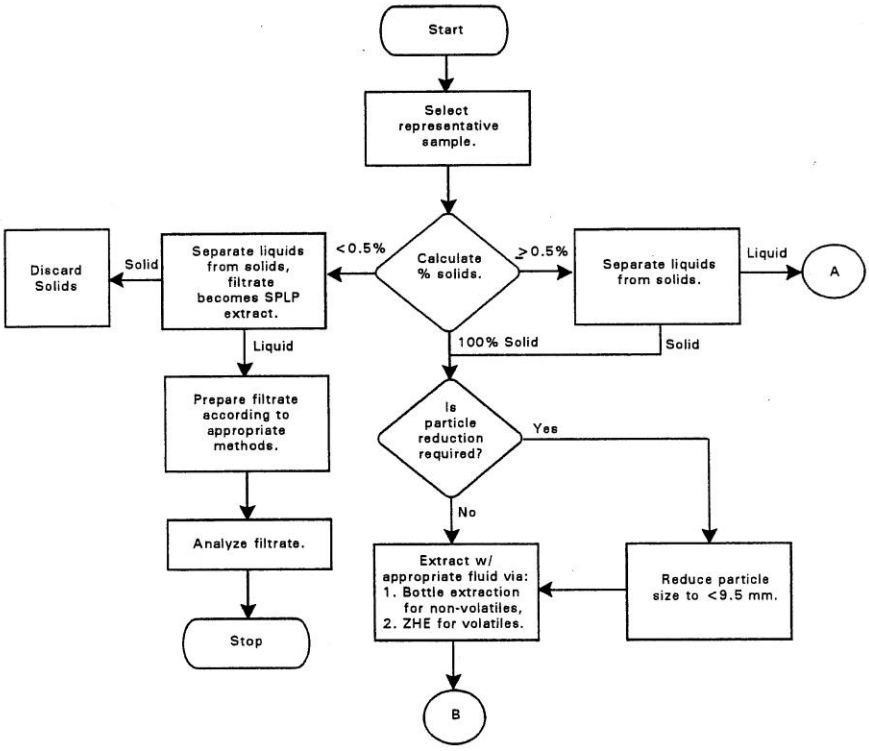
TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 2

SPLP FLOW CHARTS

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE



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Revision 0
September 1994

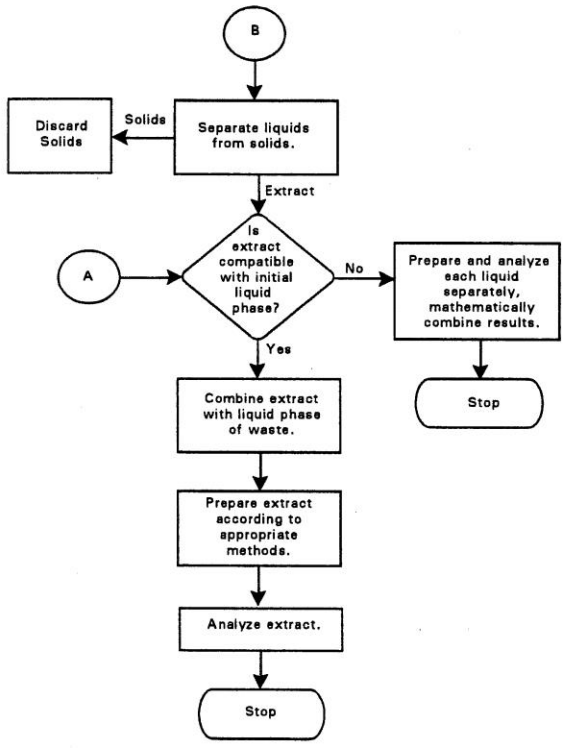
TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 3

SPLP FLOW CHARTS

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (continued)



1312 - 30

Revision 0
September 1994

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 4

SVOA SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorphenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

***** Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for SPLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 5

PESTICIDE SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)	1.0 ug/mL
Tetrachloro-m-xylene (TCMX)	1.0 ug/mL

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 6

EXAMPLE PAGE FROM SPLP FLUID USE LOGBOOK

Katahdin Analytical Services, LLC

Non-Volatile TCLP/SPLP Extraction Fluid Preparation and Use Logbook

FLUID PREPARATION

TCLP ☐ SPLP ☑	~~TCLP~~ Fluid #:	Fluid Batch #:	Prep Date:	Prepared by:	Measured pH:
	~~1391~~ SPLP 1	1391	5/22/17	MD	4.15

Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)
Glacial Acetic Acid			N.A.	MD 5/22/17
Sodium Hydroxide		N.A.		
0.6% Sulfuric Acid / 0.4% nitric acid	MR1793	2.5	N/A	20

Matrix Spike: 10mL (MS) MW17051 into 100mL Extract

FLUID USE LOG

Katahdin Sample Number	TCLP Extraction Start Date	Extract to be Analyzed for: Metals	SVOA	Pest	Herb
Blank: PBTF1391A	5/22/17	✓			
Matrix Spike: SK4095-4		✓			
SK4095-2A		✓			
SK4095-4A		✓			
SK4095-6A		✓			
SK4095-8B		✓			
SK6120-M	7-17-17	✓			

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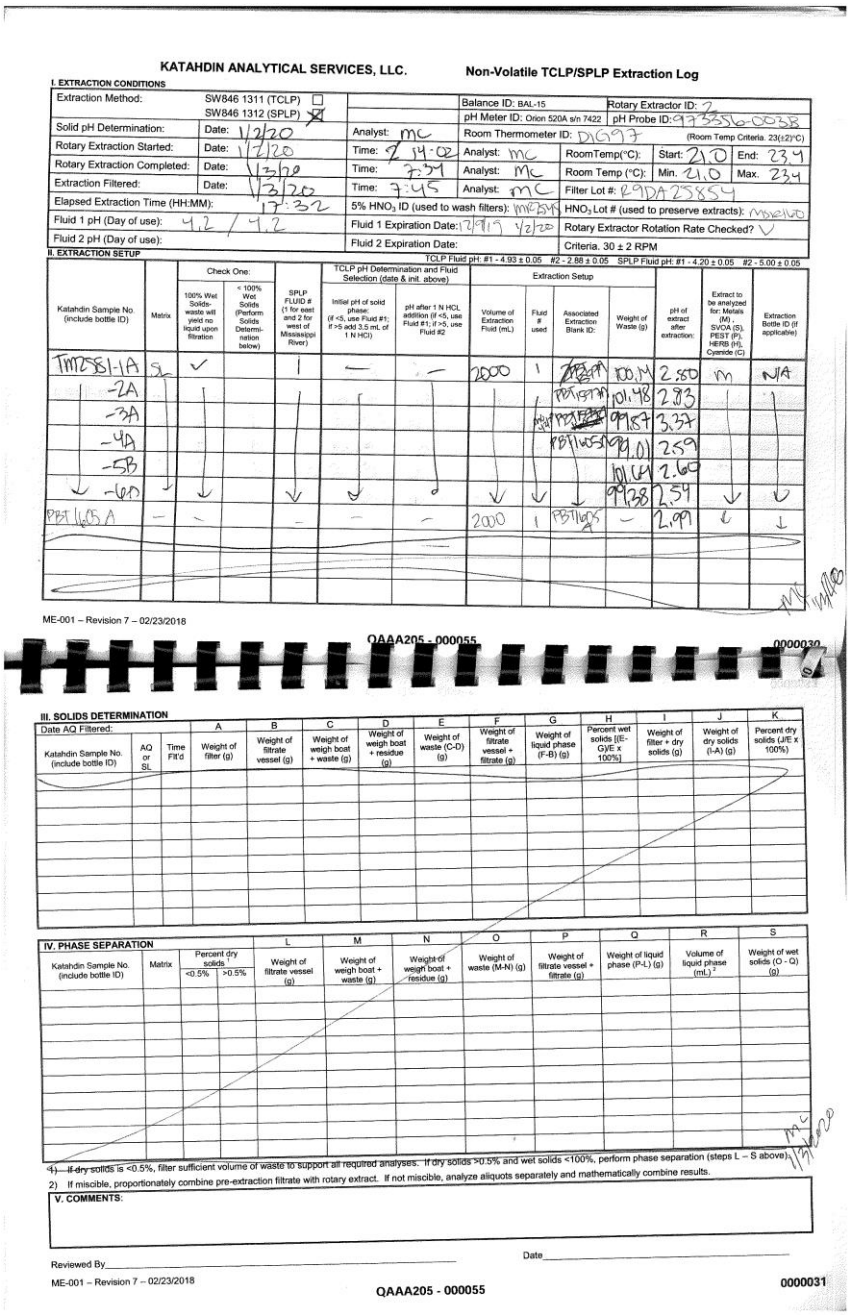
TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 7

EXAMPLE PAGE FROM NON-VOLATILE TCLP/SPLP EXTRACTION LOGBOOK



KATAHDIN ANALYTICAL SERVICES, LLC. Non-Volatile TCLP/SPLP Extraction Log

I. EXTRACTION CONDITIONS

Extraction Method:	SW846 1311 (TCLP) ☐ SW846 1312 (SPLP) ☒		Balance ID: BAL-15	Rotary Extractor ID: 2	
			pH Meter ID: Orion 520A s/n 7422	pH Probe ID: 97356-0038	
Solid pH Determination:	Date: 1/2/20	Analyst: MC	Room Thermometer ID: DIG97	(Room Temp Criteria: 23(±2)°C)	
Rotary Extraction Started:	Date: 1/2/20	Time: 7 14:02	Analyst: MC	RoomTemp(°C):	Start: 21.0 End: 23.4
Rotary Extraction Completed:	Date: 1/3/20	Time: 7:34	Analyst: MC	Room Temp (°C):	Min. 21.0 Max. 23.4
Extraction Filtered:	Date: 1/3/20	Time: 7:45	Analyst: MC	Filter Lot #: K9DA25854	
Elapsed Extraction Time (HH:MM):	17:32	5% HNO_3 ID (used to wash filters): MK25W		HNO_3 Lot # (used to preserve extracts): MKH160	
Fluid 1 pH (Day of use):	4.2 / 4.2	Fluid 1 Expiration Date: 12/9/19 1/2/20		Rotary Extractor Rotation Rate Checked? ✓	
Fluid 2 pH (Day of use):		Fluid 2 Expiration Date:		Criteria. 30 ± 2 RPM	

II. EXTRACTION SETUP — TCLP Fluid pH: #1 - 4.93 ± 0.05 #2 - 2.88 ± 0.05 SPLP Fluid pH: #1 - 4.20 ± 0.05 #2 - 5.00 ± 0.05

Katahdin Sample No. (include bottle ID)	Matrix	Check One: 100% Wet Solids- waste will yield no liquid upon filtration	Check One: < 100% Wet Solids (Perform Solids Determination below)	SPLP FLUID # (1 for east and 2 for west of Mississippi River)	TCLP pH Determination and Fluid Selection (date & init. above): Initial pH of solid phase: (if <5, use Fluid #1; if >5 add 3.5 mL of 1 N HCl)	pH after 1 N HCL addition (if <5, use Fluid #1; if >5, use Fluid #2	Extraction Setup: Volume of Extraction Fluid (mL)	Fluid # used	Associated Extraction Blank ID:	Weight of Waste (g)	pH of extract after extraction:	Extract to be analyzed for: Metals (M) , SVOA (S), PEST (P), HERB (H), Cyanide (C)	Extraction Bottle ID (if applicable)
TM2581-1A	SL	✓		1	—	—	2000	1	PBTM25W	100.14	2.80	M	N/A
-2A									PBTM25W	101.48	2.83		
-3A									PBTM25W	99.57	3.37		
-4A									PBTM25W	99.01	2.59		
-5B										101.64	2.60		
-6B										99.38	2.54		
PBTM25A	—	—	—	—	—	—	2000	1	PBTM25W	—	2.99		

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III. SOLIDS DETERMINATION

Date AQ Filtered:

Katahdin Sample No. (include bottle ID)	AQ or SL	Time Filt'd	A Weight of filter (g)	B Weight of filtrate vessel (g)	C Weight of weigh boat + waste (g)	D Weight of weigh boat + residue (g)	E Weight of waste (C-D) (g)	F Weight of filtrate vessel + filtrate (g)	G Weight of liquid phase (F-B) (g)	H Percent wet solids [(E-G)/E x 100%]	I Weight of filter + dry solids (g)	J Weight of dry solids (I-A) (g)	K Percent dry solids (J/E x 100%)

IV. PHASE SEPARATION

Katahdin Sample No. (include bottle ID)	Matrix	Percent dry solids[1] <0.5%	Percent dry solids[1] >0.5%	L Weight of filtrate vessel (g)	M Weight of weigh boat + waste (g)	N Weight of weigh boat + residue (g)	O Weight of waste (M-N) (g)	P Weight of filtrate vessel + filtrate (g)	Q Weight of liquid phase (P-L) (g)	R Volume of liquid phase (mL)[2]	S Weight of wet solids (O - Q) (g)

[1] If dry solids is <0.5%, filter sufficient volume of waste to support all required analyses. If dry solids >0.5% and wet solids <100%, perform phase separation (steps L – S above).

2) If miscible, proportionately combine pre-extraction filtrate with rotary extract. If not miscible, analyze aliquots separately and mathematically combine results.

V. COMMENTS:

Reviewed By_____ Date_____

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ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: G. Brewer

Review Date: 01/19/21

SOP Number: CA-620-07

SOP Title: SPLP for Inorganic and Non-Volatile Organic Analytes

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: G. Brewer **Date:** 01/19/21

QAO Signature: Leslie Dimond **Date:** 012521

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Jol C Butler Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03·27·01

General Manager: Dennis P. Hoffman Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. Changed Run ID naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tubing size Sect. 5 - changed acid conc.'s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated Tables 1, 3 → 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards. Updated Table 1 with Serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.1.8 - changed instrument identifier to reflect new instrument; section 8 - changed acceptance criteria and ICSAB analyte list; Table 1 - updated acceptance criteria and corrective actions to QC. Table 3 - added all analytes to list - removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 6020A. Added Hardness by calculation attachment. Added LLOQ requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 3.4.1, 4.2, 5.2 7.9, 7.10, 7.11, 7.16 and 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect. 10 - Added, ~~edited references~~. Updated Tables 1, 5, 6, 7, 8 and 9 edited references LAD 042512	LAD	04/12	04/12
09	Sect. 7 - Added reference to autosampler software, added printing calibration and removed printing of run summary	LAD	08/13	08/13
10	Sect. 7 - Updated for changes made in the Metals database for importing and handling data. Sect. 10 - updated and added references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 7, Table 1, 2, 3, 6, 8 & 11 - Updated to reflect change from 5 pt. to 2 pt. calibration. Table 7, 8 & 9 - Updated to reflect change in Aluminum PQL	LAD	04/16	04/16
12	Change title of Section 5.0. Update method references for NELAC + DoD. Minor additions/corrections to Sections 3.0, 4.2, 7.3.5 and table 5.	LAD	09/17	09/17
13	Table 1 - Added ms/msD, corrected section references - Table 2 - Removed, DoD QSM 4.2 QC Requirements. Renumbered subsequent sections. Table 7 - Added Thorium - Updated Table references throughout SOP	LAD	01/19	01/19
14	Sect.s 4, 7, 8 and 10 updated for the new Agilent 7800 ICPMS System and the Mass Hunter Software.	LAD	06/20	06/20

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
15	Corrected typos. Modified Section 7.19 to reflect current practice. Modified Tables 5, 6, 7 to reflect current practice. Added discussion of collision cell to Sections 3 and 4.1, and modified Table 14 to include reaction gases.	Lesie Dimond	02/21	02/21

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-15**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-15**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions.
If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7800 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7800 uses a collision cell to remove most isobaric interferences. In the collision cell, a non-reactive gas (hydrogen or helium) is introduced under pressure in the path of the plasma gas. The non-reactive gas selectively attenuates all polyatomic interferences based on their size. The process exploits the fact that all polyatomic ions are larger than analyte ions of the same mass, so they collide with the cell gas more often as they pass through the cell, emerging with lower residual energy. These low energy ions are excluded from the ion beam entering the spectrometer by a bias voltage at the cell exit. The collision gas that is used for each element is listed in Table 14.

The Agilent 7800 MassHunter data system is also used to correct for these polyatomic interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) = (m/z 75 signal) - (2.95) (m/z 77 signal) + (2.548) (m/z 82 signal) – (2.571) (m/z 83 signal), where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).

Similarly:
Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.84)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 4. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7800 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See Table 14 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

4.1 Agilent 7800 ICP-MS system, consisting of the Agilent 7800 ICP-mass spectrometer and its controlling computer data station. The quadropole spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7800 mass range of 2-260 amu exceeds the method requirement of 2-240 amu. The instrument is equipped with an octopole collision cell that significantly reduces polyatomic isobaric interferences. The Agilent 7800 MassHunter software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX Fast Autosampler and Sample Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 3-stop PVC flared black-black (0.76 mm ID) and orange-green-orange (0.38 mm ID). 3-stop ESI PVC flared yellow-blue (1.52 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon, hydrogen, and helium gas supplies (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area designed for trace element sample handling must be used. Standards, samples, and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker "Instra-Analyzed" trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals and supplied by the vendors with certificates of purity and analysis. Refer to Tables 6 and 7 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 6 for a listing of intermediate standards required and for preparation instructions. Refer to Table 8 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards

Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCI for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent MassHunter software.

7.2 Initialize the autosampler by opening “ESI SC” located on the Desktop and click “Initialize.” Wait for the autosampler to initialize and the COM port to be opened, indicated by the text color changing to GREEN.

7.3 The main MassHunter software is accessed by double-clicking the “ICP-MS Instrument Control” icon on the Desktop.

7.4 Check argon gas pressure (>100 psi).

7.5 Turn on the water chiller/recirculator.

7.6 Verify that the exhaust hood is in operation.

7.7 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.8 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.9 Verify that the drain reservoir has adequate room to accept the day’s drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.10 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.11 Move the carrier and internal standard probe to a blank solution.

7.12 Open the MassHunter software by double-clicking the “ICP-MS Top” icon. Initiate the plasma by selecting Plasma>>Plasma On>>Okay and allow the instrument to aspirate calibration blank solution for at least 10 minutes. During this warm-up, select Start Signal Monitor to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.13 Move the carrier line to the Agilent Tune Solution (ATS). Verify there is ATS in the ATS autosampler tube. Increase the nebulizer pump speed to quickly pump solution through the lines. Verify there are no air bubbles in the lines by allowing a large bubble to pass through both the carrier and internal standard lines followed by the previously mentioned solutions.

7.14 After the warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.15 Open a new batch by selecting Batch>>Open Batch from Template>> 1PT_Cal_ORS. Rename the batch to the meet standard format for ICP-MS runs. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("L" for the Agilent 78000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("G" for 2013, "H" for 2014, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "LME01A" is the first run of the day that was performed on May, 1, 2019, using the Agilent 7800 ICP-MS.

7.16 Generate a tune report by selecting Acq Method>>Tune>>Perform Auto Tune>> All Tune Modes. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.19.

7.17 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting Tune>>Autotune>>Run. The MassHunter software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 13.

7.18 Repeat step 7.16 until all tune specifications have been met. File the final tune report.

7.19 Once the tune report has been generated and confirmed to be acceptable, save the tune report. Change the nebulizer pump speed to "fast", move the carrier probe into the carrier solution, and move the internal standard probe into the internal standard solution. Allow a large air bubble to move through each line, followed by the solutions. Once the solutions reach the rotor, change the nebulizer pump speed to "normal".

7.20 Edit the sequence template to create an analytical sequence table listing all of the samples to be analyzed. Fill in the sample table with sample type, sample IDs, vial numbers, dilution factors.

7.21 Load the autosampler racks according to the analytical sequence.

7.22 Select Add to QUEUE. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate

frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least one multielement calibration standard to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.23 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.24 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.25 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.26 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.27 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.6, 8.7, and 8.9. The sample must be reanalyzed for the element in question.

7.28 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.29 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.30 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to

compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.31 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.32 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.33 Follow these steps to create the data import file: From the Online (or Offline) ICP-MS Data Analysis window, open the specific batch to be imported to the database.

7.34 Once opened, use the number column (furthest to the left) to highlight all samples to be imported. Click and drag down, or alternatively click, shift, scroll and click. Always include two rinses after the closing PQL in the import file to ensure proper database functionality.

7.35 Once all samples are highlighted, click the down-facing arrow next to the “Report” icon at the top of the ICP-MS Data Analysis window, click “LIMS – Export Selected Samples,” and wait for a text file to open and notification to appear. Close the text file and notification. The text file that appears is the import file to be imported into the database.

To import data into the Metals Database:

7.36 Open the Metals Database "**KIMS_Metals**" from the Desktop.

7.37 Click "**Data Import Menu**," then **enter analyst initials** and in this order click: "**Agilent 7800**," and "**Import and Validate Run**."

7.38 In the window that appears, in this order, click: "**Desktop**", "**metals on Katahdin File Server (Server_a)**," "**L-ICPMSDATA**." From here, open the text file created in the process above (i.e. "LNF09A.txt").

7.39 Allow all queries to run and wait for batch QC results to appear. Uncheck the boxes next to all failing batch QC results. Click away from the last unchecked box to finalize the selection. Click "**Accept Selected Batch QC**."

7.40 Minimize the main menu form. In the window **"KIMS_Metals : Database**," Click the header: "**Queries**," and find the query "**transfer requested data from archive to instr data**." To the right, click "**Design**." The fourth column in the window that opens should have the header called "**File**." Find that column, and under it in the row that calls for "**Criteria**," type the file name you just imported (i.e. "LNF09A"). Close and save this window. Note: This window can be used to filter by many other criteria as well.

7.41 Review and accept or reject the data in the table "**Archive Data**" based on the QC limits in the Table 1 and Table 2 of the following sections.

7.42 Restore the main menu.

7.43 Click "**Additional Data Settings**"

7.44 Click "**Report Added Compounds**"

7.45 Accept the data that comes through. If this process takes more than five minutes, click only once and DO NOT HOLD **Ctrl + Break**. Once the main menu reappears, open the "**Instrument Data**" table and delete its contents.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards

may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least one multielement calibration standard. For each element,

calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted and reanalyzed. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples analyzed are for the Department of Defense (80% to 120% recovery limits) or other

client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as “estimated” with a “J”.

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations,

and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

8.12 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12.1 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12.2 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.16 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

8.17 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the

contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version 5.3.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7800/7900 ICP-MS Hardware Maintenance Manual, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS MassHunter Workstation Intelligent Sequence Software, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS Familiarization Guide, Agilent Technologies, Inc., 2018.

Agilent 7800 ICP-MS(7800/7900) MassHunter Workstation User Guide, Agilent Technologies, Inc., 2018.

List of Tables

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	If more than 1 calibration std is used, correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit, or if sample result > 10x detected PQL concentration
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements, Unless ICSAB passes and trace metal element is > 10x absolute value of ICSA trace-metals concentration.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1 (continued)

QC REQUIREMENTS

Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.
Matrix Spike Sample (S), or Matrix Spike Duplicate Sample (P)	At least one per digestion batch of 20 or fewer samples.	Recovery within ±25% of true value, if sample <4x spike added.	Flag results
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.13	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 30%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	Daily.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration = 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICALhas passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL	If recoveries are acceptable for QC samples, but not field samples, the field	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		blank.	samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.		should be selected.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > ½ LOQ.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. Flagging is not appropriate.	Results may not be reported without a valid calibration blank.	For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < ½ LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory	A laboratory must use the QSM	Examine the project-specific	For the specific analyte(s) in the	If MS results are outside the limits, the

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
	batch.	Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	requirements. Contact the client as to additional measures to be taken.	parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-14	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: $\pm$ PQL	Acceptance criteria stated in 6020A: <3 times IDL

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TABLE 4

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	$^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571$ [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	$^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764$ [See note 3] $^{114}Cd = (114)*1 – (118)*0.0268 – (108)*1.84$ [See note 4]
	Calcium	Ca	44	$^{44}Ca = (44)*1 – (88)*0.0169$ [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	$^{54}Fe = (54)*1 – (52)*0.0282$ [See note 8] $^{57}Fe = (57)*1 – (43)*0.03$ [See note 9]
	Lead	Pb	206, 207, 208	$^{208}Pb = (208)*1 + (206)*1 + (207)*1$ [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	$^{98}Mo = (98)*1 – (99)*0.146$ [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	$^{82}Se = (82)*1 – (83)*1.009$ [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 – (53)*2.95 + (52)*0.378$ [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	$^{115}In = (115)*1 – (118)*0.016$ [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 4 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1, CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B + W Solution (0.5mL each per 50mL and use same day only)	Lab Prepared	0.200
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Stock Tuning Solution	Agilent	0.025
	Conc. HNO_3	Baker Instra Analyzed	1
	Conc HCl	Baker Instra Analyzed	0.5
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10
	Conc HNO3	Baker Instra Analyzed	4.0
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (5% HNO_2/5%HCL)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Al, Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Th, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 (5% HNO_2/5%HCL)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
	Conc. HCL	Baker Instra Analyzed	2
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TABLE 6 (Continued)

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each
	1000mg/L Pb		0.30
1000 mg/L B + W Solution (prepare on day of use	1000 mg/L B	Inorganic Ventures	1.0
	1000 mg/L W		1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	CCV	Cal. Std.	ICV	PQL	
Aluminum	500.0	1000.0	400.0	20.0	
Antimony	25.0	50.0	20.0	0.2	
Arsenic	25.0	50.0	20.0	1.0	
Barium	25.0	50.0	20.0	0.4	
Beryllium	25.0	50.0	20.0	0.2	
Boron	25.0	50.0	20.0	4.0	
Cadmium	25.0	50.0	20.0	0.2	
Calcium	5000.0	10000.0	4000.0	20.0	
Chromium	25.0	50.0	20.0	1.0	
Cobalt	25.0	50.0	20.0	0.2	
Copper	25.0	50.0	20.0	0.6	
Iron	5000.0	10000.0	4000.0	20.0	
Lead	25.0	50.0	20.0	0.2	
Magnesium	5000.0	10000.0	4000.0	20.0	
Manganese	25.0	50.0	20.0	0.4	
Molybdenum	25.0	50.0	40.0	1.0	
Nickel	25.0	50.0	20.0	0.4	
Potassium	5000.0	10000.0	4000.0	200.0	
Selenium	25.0	50.0	20.0	1.0	
Silicon	500.0	1000.0	400.0	100.0	
Silver	25.0	50.0	20.0	0.2	
Sodium	5000.0	10000.0	4000.0	200.0	
Strontium	25.0	50.0	20.0	1.0	
Thallium	25.0	50.0	20.0	0.2	
Thorium	25.0	50.0	20.0	0.2	
Tin	25.0	50.0	20.0	1.0	
Tungsten	25.0	50.0	20.0	1.0	
Uranium	25.0	50.0	20.0	0.2	
Vanadium	25.0	50.0	20.0	1.0	
Zinc	25.0	50.0	20.0	2.0	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Thorium		20			
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Cerium					10.0
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	2.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	3.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Thorium		0.02			0.2	
Tungsten	5.0	0.10				0.2
Uranium		0.02			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 11

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2^{o} C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 12

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 13

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 14

REPORTED ISOTOPES, INTERNAL STANDARDS, AND COLLISION CELL GASES

ELEMENT	MASS	INTERNAL STANDARD (mass)	COLLISION CELL GAS
Aluminum	27	Scandium (45)	No Gas
Antimony	123	Terbium (159)	No Gas
Arsenic	75	Yttrium (89)	Helium
Barium	135	Terbium (159)	No Gas
Beryllium	9	Lithium (6)	No Gas
Boron	11	Lithium (6)	No Gas
Cadmium	114	Yttrium (89)	Helium
Calcium	44	Scandium (45)	Hydrogen
Chromium	52	Yttrium (89)	Helium
Cobalt	59	Yttrium (89)	Helium
Copper	65	Yttrium (89)	Helium
Iron	57	Yttrium (89)	Hydrogen
Lead	208	Bismuth (209)	No Gas
Magnesium	25	Scandium (45)	Helium
Manganese	55	Yttrium (89)	Helium
Molybdenum	98	Yttrium (89)	Helium
Nickel	60	Yttrium (89)	Helium
Potassium	39	Scandium (45)	Helium
Selenium	82	Yttrium (89)	Hydrogen
Silicon	29	Scandium (45)	Hydrogen
Silver	107	Yttrium (89)	Helium
Sodium	23	Scandium (45)	Helium
Strontium	88	Yttrium (89)	Helium
Thallium	203	Bismuth (209)	No Gas
Thorium	232	Bismuth (209)	No Gas
Tin	118	Terbium (159)	Helium
Tungsten	182	Terbium (159)	No Gas
Uranium	238	Bismuth (209)	No Gas
Vanadium	51	Yttrium (89)	Helium
Zinc	66	Yttrium (89)	Helium

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in “Standard Methods for the Examination if Water and Wastewater,” Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

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 V5.4)

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 2025
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 5.4)

Katahdin Analytical Services, LLC

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

TESTING

Valid to: **February 1, 2025** 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

Non-Potable Water		
Technology	**Method**	**Analyte**
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	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)

Non-Potable Water		
Technology	**Method**	**Analyte**
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)
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)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
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	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 9056A	Bromide
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
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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)
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)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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)
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)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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	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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
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	3,4-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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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)
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
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	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9071A/B	Oil and Grease, Oil and Grease with SGT
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
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
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Sieves, Hydrometer	ASTM D422	Grain Size
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

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
Organic Preparation	EPA 8330A/B	ISM

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
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

Biological Tissue		
Technology	**Method**	**Analyte**
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)
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

Biological Tissue		
Technology	**Method**	**Analyte**
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
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

Biological Tissue		
Technology	**Method**	**Analyte**
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
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

Biological Tissue		
Technology	**Method**	**Analyte**
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
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

Biological Tissue		
Technology	**Method**	**Analyte**
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	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

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Alpha Analytical, Inc.
320 Forbes Blvd.
Mansfield, MA 02048

Fulfills the requirements of

ISO/IEC 17025:2017

and the

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: 30 May 2023
Certificate Number: L2474

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 V5.3)

Alpha Analytical, Inc.
320 Forbes Blvd.
Mansfield, MA 02048
James Todaro
508-898-9220

TESTING

Valid to: **May 30, 2023** Certificate Number: **L2474**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Hexachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Heptachlorobiphenyls

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Decachlorobiphenyl
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) CAS# 1746-01-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD) CAS# 40321-76-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS#39227-28-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 57653-85-7
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 19408-74-3
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD) CAS# 35822-46-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8,9 -Octachlorodibenzo-p-dioxin (OCDD) CAS#3268-87-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran (TCDF) CAS# 51207-31-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-41-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-31-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 70648-26-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS#57117-44-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF) CAS#72918-21-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 60851-34-5
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF) CAS# 67562-39-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF) CAS# 55673-89-7
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8,9 -Octachlorodibenzofuran (OCDF) CAS#39001-02-0
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Tetrachlorodibenzo-p-dioxin (TCDD) CAS#41903-57-5

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Pentachlorodibenzo-p-dioxin (PeCDD) CAS#36088-22-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Hexachlorodibenzo-p-dioxin (HxCDD) CAS#34465-46-8
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Heptachlorodibenzo-p-dioxin (HpCDD) CAS#37871-00-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Tetrachlorodibenzofuran (TCDF) CAS#55722-27-5
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Pentachlorodibenzofuran (PeCDF) CAS#30402-15-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Hexachlorodibenzofuran (HxCDF) CAS#55684-94-1
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Heptachlorodibenzofuran (HpCDF) CAS#38998-75-3
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total PCDF
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total PCDD
GC/MS-SIM	EPA 8270E-SIM Isotope Dilution	1,4-Dioxane
SPE/LC/MS/MS	Alpha SOP #29033[2]	N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) (cas# 2991-50-6)
SPE/LC/MS/MS	Alpha SOP #29033[2]	N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) (cas# 2355-31-9)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorobutanesulfonic acid (PFBS) (cas# 375-73-5)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorodecanoic acid (PFDA) (cas# 335-76-2)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorododecanoic acid (PFDoA) (cas# 307-55-1)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluoroheptanoic acid (PFHpA) (cas# 375-85-9)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorohexanesulfonic acid (PFHxS) (cas# 355-46-4)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorohexanoic acid (PFHxA) (cas# 307-24-4)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorononanoic acid (PFNA) (cas# 375-95-1)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorooctanesulfonic acid (PFOS) (cas# 1763-23-1)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorooctanoic acid (PFOA) (cas# 335-67-1)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorotetradecanoic acid (PFTA) (cas# 376-06-7)

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluorotridecanoic acid (PFTrDA) (cas# 72629-94-8)
SPE/LC/MS/MS	Alpha SOP #29033[2]	Perfluoroundecanoic acid (PFUnA) (cas# 2058-94-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethyl perfluorooctanesulfonamidoacetic acid N-EtFOSAA (cas# 2991-50-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methyl perfluorooctanesulfonamidoacetic acid N-MeFOSAA (cas# 2355-31-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanesulfonic acid (PFBS) (cas# 375-73-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanoic acid PFDA (cas# 335-76-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanoic acid PFDoA (cas# 307-55-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanoic acid PFHpA (cas# 375-85-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanesulfonic acid (PFHxS) (cas# 355-46-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanoic acid PFHxA (cas# 307-24-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanoic acid PFNA (cas# 375-95-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonic acid (PFOS) (cas# 1763-23-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanoic acid PFOA (cas# 335-67-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotridecanoic acid PFTrDA (cas# 72629-94-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroundecanoic acid PFUnA (cas# 2058-94-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotetradecanoic acid PFTA (cas# 376-06-7)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanoic acid (PFPeA) (cas# 2706-90-3)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanoic acid PFBA (cas# 375-22-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanesulfonic acid (PFDS) (cas# 335-77-3)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanesulfonic acid (PFNS) (cas# 68259-12-1)

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanesulfonic acid (PFHpS) (cas# 375-92-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanesulfonic acid (PFPeS) (cas# 2706-91-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonamide PFOSA (cas# 754-91-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorodecane sulfonic acid (8:2) 8:2FTS (cas# 39108-34-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorooctane sulfonic acid (6:2) 6:2FTS (cas# 27619-97-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2) 4:2FTS (cas# 757124-72-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid (PFDoS) (cas# 79780-39-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) (cas# 763051-92-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) (cas# 756426-58-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamide (NMeFOSA) (cas# 31506-32-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamide (NEtFOSA) (cas# 4151-50-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2) (10:2FTS) (cas# 120226-60-0)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMeFOSE) (cas# 24448-09-7)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtFOSE) (cas# 1691-99-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid PFHxDA (cas# 67905-19-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctadecanoic acid PFODA (cas# 16517-11-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Tetrafluoro-2(heptafluoropropoxy)propanoic acid HFPO-DA (cas# 13252-13-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4,8-dioxa-3H-perfluorononanoic acid ADONA (cas# 919005-14-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA) 377-73-1
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropropanesulfonic acid (PFPrS) 423-41-6
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) 763051-92-9
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) 756426-58-1
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Hexafluoropropylene oxide dimer acid (HFPO-DA) 13252-13-6
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorobutanoic acid (PFBA) 375-22-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorobutanesulfonic acid (PFBS) 375-73-5
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) 39108-34-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorodecanoic acid (PFDA) 335-76-2
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorododecanoic acid (PFDoA) 307-55-1
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoroheptanesulfonic acid (PFHpS) 375-92-8
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoroheptanoic acid (PFHpA) 375-85-9
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	1H,1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2FTS) 757124-72-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorohexanesulfonic acid (PFHxS) 355-46-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorohexanoic acid (PFHxA) 307-24-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoro-3-methoxypropanoic acid (PFMPA) 377-73-1
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorononanoic acid (PFNA) 375-95-1
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) 27619-97-2
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorooctanesulfonic acid (PFOS) 1763-23-1

Non-Potable Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluorooctanoic acid (PFOA) 335-67-1
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoropentanoic acid (PFPeA) 2706-90-3
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoropentanesulfonic acid (PFPeS) 2706-91-4
SPE/LC/MS/MS Isotope Dilution	Alpha SOP #36216[2]	Perfluoroundecanoic acid (PFUnA) 2058-94-8
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl(2,2',3,3',5-)(PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668A	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668A	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Decachlorobiphenyl
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl(2,2',3,3',5-)(PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668C	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668C	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Decachlorobiphenyl
Preparation	**Method**	**Type**
Extraction	EPA 3510C	Separatory Funnel
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS, SIM	EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) CAS# 1746-01-6

Drinking Water		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 522	1,4-Dioxane
SPE/LC/MS/MS Isotope Dilution	EPA 533	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) 763051-92-9
SPE/LC/MS/MS Isotope Dilution	EPA 533	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) 756426-58-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Hexafluoropropylene oxide dimer acid (HFPO-DA) 13252-13-6
SPE/LC/MS/MS Isotope Dilution	EPA 533	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorobutanoic acid (PFBA) 375-22-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorobutanesulfonic acid (PFBS) 375-73-5
SPE/LC/MS/MS Isotope Dilution	EPA 533	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) 39108-34-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorodecanoic acid (PFDA) 335-76-2
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorododecanoic acid (PFDoA) 307-55-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoroheptanesulfonic acid (PFHpS) 375-92-8
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoroheptanoic acid (PFHpA) 375-85-9
SPE/LC/MS/MS Isotope Dilution	EPA 533	1H,1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2FTS) 757124-72-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorohexanesulfonic acid (PFHxS) 355-46-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorohexanoic acid (PFHxA) 307-24-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoro-3-methoxypropanoic acid (PFMPA) 377-73-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorononanoic acid (PFNA) 375-95-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) 27619-97-2
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorooctanesulfonic acid (PFOS) 1763-23-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluorooctanoic acid (PFOA) 335-67-1
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoropentanoic acid (PFPeA) 2706-90-3
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoropentanesulfonic acid (PFPeS) 2706-91-4
SPE/LC/MS/MS Isotope Dilution	EPA 533	Perfluoroundecanoic acid (PFUnA) 2058-94-8

Drinking Water		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	EPA 537.1	N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) (cas# 2991-50-6)
SPE/LC/MS/MS	EPA 537.1	N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) (cas# 2355-31-9)
SPE/LC/MS/MS	EPA 537.1	Perfluorobutanesulfonic acid (PFBS) (cas# 375-73-5)
SPE/LC/MS/MS	EPA 537.1	Perfluorodecanoic acid (PFDA) (cas# 335-76-2)
SPE/LC/MS/MS	EPA 537.1	Perfluorododecanoic acid (PFDoA) (cas# 307-55-1)
SPE/LC/MS/MS	EPA 537.1	Perfluoroheptanoic acid (PFHpA) (cas# 375-85-9)
SPE/LC/MS/MS	EPA 537.1	Perfluorohexanesulfonic acid (PFHxS) (cas# 355-46-4)
SPE/LC/MS/MS	EPA 537.1	Perfluorohexanoic acid (PFHxA) (cas# 307-24-4)
SPE/LC/MS/MS	EPA 537.1	Perfluorononanoic acid (PFNA) (cas# 375-95-1)
SPE/LC/MS/MS	EPA 537.1	Perfluorooctanesulfonic acid (PFOS) (cas# 1763-23-1)
SPE/LC/MS/MS	EPA 537.1	Perfluorooctanoic acid (PFOA) (cas# 335-67-1)
SPE/LC/MS/MS	EPA 537.1	Perfluorotetradecanoic acid (PFTA) (cas# 376-06-7)
SPE/LC/MS/MS	EPA 537.1	Perfluorotridecanoic acid (PFTrDA) (cas# 72629-94-8)
SPE/LC/MS/MS	EPA 537.1	Perfluoroundecanoic acid (PFUnA) (cas# 2058-94-8)
SPE/LC/MS/MS	EPA 537.1	Hexafluoropropylene oxide dimer acid (HFPA-DA) (cas# 13252-13-6)
SPE/LC/MS/MS	EPA 537.1	11-chloroeicosafluoro-3- oxaundeacane -1-sulfonic acid (11Cl-PF3OUdS) (cas# 763051-92-9)
SPE/LC/MS/MS	EPA 537.1	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) (cas# 756426-58-1)
SPE/LC/MS/MS	EPA 537.1	4,8-dioxa-3H-perfluorononanoic acid (ADONA) (cas# 919005-14-4)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl(2,2',3,3',5-)(PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668A	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668A	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Tetrachlorobiphenyls

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Decachlorobiphenyl
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)

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Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl(2,2',3,3',5-)(PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)

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Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)

Drinking Water		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668C	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668C	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Decachlorobiphenyl

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Hexachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Heptachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Decachlorobiphenyl
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) CAS# 1746-01-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD) CAS# 40321-76-4

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS#39227-28-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 57653-85-7
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 19408-74-3
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD) CAS# 35822-46-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8,9 -Octachlorodibenzo-p-dioxin (OCDD) CAS#3268-87-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran (TCDF) CAS# 51207-31-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-41-6
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-31-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 70648-26-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS#57117-44-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF) CAS#72918-21-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 60851-34-5
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF) CAS# 67562-39-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF) CAS# 55673-89-7
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	1,2,3,4,6,7,8,9 -Octachlorodibenzofuran (OCDF) CAS#39001-02-0
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Tetrachlorodibenzo-p-dioxin (TCDD) CAS#41903-57-5
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Pentachlorodibenzo-p-dioxin (PeCDD) CAS#36088-22-9
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Hexachlorodibenzo-p-dioxin (HxCDD) CAS#34465-46-8
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Heptachlorodibenzo-p-dioxin (HpCDD) CAS#37871-00-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Tetrachlorodibenzofuran (TCDF) CAS#55722-27-5

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Pentachlorodibenzofuran (PeCDF) CAS#30402-15-4
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Hexachlorodibenzofuran (HxCDF) CAS#55684-94-1
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total Heptachlorodibenzofuran (HpCDF) CAS#38998-75-3
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total PCDF
GC/Hi-Res MS, SIM	EPA 8290A / EPA 1613B	Total PCDD
Gravimetric	SM 2540G	Percent Total Solids
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethyl perfluorooctanesulfonamidoacetic acid N-EtFOSAA (cas# 2991-50-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methyl perfluorooctanesulfonamidoacetic acid N-MeFOSAA (cas# 2355-31-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanesulfonic acid (PFBS) (cas# 375-73-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanoic acid PFDA (cas# 335-76-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanoic acid PFDoA (cas# 307-55-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanoic acid PFHpA (cas# 375-85-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanesulfonic acid (PFHxS) (cas# 355-46-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanoic acid PFHxA (cas# 307-24-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanoic acid PFNA (cas# 375-95-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonic acid (PFOS) (cas# 1763-23-1)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanoic acid PFOA (cas# 335-67-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotridecanoic acid PFTrDA (cas# 72629-94-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroundecanoic acid PFUnA (cas# 2058-94-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotetradecanoic acid PFTA (cas# 376-06-7)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentatonic acid (PFPeA) (cas# 2706-90-3)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanoic acid PFBA (cas# 375-22-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluordecanesulfonic acid (PFDS) (cas# 335-77-3)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanesulfonic acid (PFNS) (cas# 68259-12-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanesulfonic acid (PFHpS) (cas# 375-92-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanesulfonic acid (PFPeS) (cas# 2706-91-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonamide PFOSA (cas# 754-91-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorodecane sulfonic acid (8:2) 8:2FTS (cas# 39108-34-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorooctane sulfonic acid (6:2) 6:2FTS (cas# 27619-97-2)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2) 4:2FTS (cas#757124-72-4)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid (PFDoS) (cas# 79780-39-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) (cas# 763051-92-9)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) (cas# 756426-58-1)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-methylperfluoro-1-octanesulfonamide (NMeFOSA) (cas# 31506-32-8)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-ethylperfluoro-1-octanesulfonamide (NEtFOSA) (cas# 4151-50-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2) (10:2FTS) (cas# 120226-60-0)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMeFOSE) (cas# 24448-09-7)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtFOSE) (cas# 1691-99-2)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid PFHxDA (cas# 67905-19-5)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroocatdecanoic acid PFODA (cas# 16517-11-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Tetrafluoro-2-(heptafluoropropoxy)propanoic acid HFPO-DA (cas# 13252-13-6)
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4,8-dioxa-3H-perfluorononanoic acid ADONA (cas# 919005-14-4)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA) 377-73-1
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6
SPE/LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropropanesulfonic acid (PFPrS) 423-41-6
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,2',3,3',5-) (PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668A	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668A	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Decachlorobiphenyl
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,2',3,3',5-) (PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668C	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668C	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Hexachlorobiphenyls

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Decachlorobiphenyl
Preparation	**Method**	**Type**
Extraction	EPA 3570	Microscale Extraction (MSE)
Waste Dilution	EPA 3580A	Waste Dilution
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Monochlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Dichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Trichlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Tetrachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Pentachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Hexachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Heptachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Octachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Nonachlorobiphenyls
GC/MS-SIM	EPA 8270E-SIM / EPA 680	Decachlorobiphenyl
GC/Hi-Res MS, SIM	EPA 8290A	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) CAS# 1746-01-6
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD) CAS# 40321-76-4
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS#39227-28-6
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 57653-85-7
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD) CAS# 19408-74-3
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD) CAS# 35822-46-9

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,6,7,8,9 -Octachlorodibenzo-p-dioxin (OCDD) CAS#3268-87-9
GC/Hi-Res MS, SIM	EPA 8290A	2,3,7,8-Tetrachlorodibenzofuran (TCDF) CAS# 51207-31-9
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-41-6
GC/Hi-Res MS, SIM	EPA 8290A	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF) CAS#57117-31-4
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 70648-26-9
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS#57117-44-9
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF) CAS#72918-21-9
GC/Hi-Res MS, SIM	EPA 8290A	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF) CAS# 60851-34-5
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF) CAS# 67562-39-4
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF) CAS# 55673-89-7
GC/Hi-Res MS, SIM	EPA 8290A	1,2,3,4,6,7,8,9 -Octachlorodibenzofuran (OCDF) CAS#39001-02-0
GC/Hi-Res MS, SIM	EPA 8290A	Total Tetrachlorodibenzo-p-dioxin (TCDD) CAS#41903-57-5
GC/Hi-Res MS, SIM	EPA 8290A	Total Pentachlorodibenzo-p-dioxin (PeCDD) CAS#36088-22-9
GC/Hi-Res MS, SIM	EPA 8290A	Total Hexachlorodibenzo-p-dioxin (HxCDD) CAS#34465-46-8
GC/Hi-Res MS, SIM	EPA 8290A	Total Heptachlorodibenzo-p-dioxin (HpCDD) CAS#37871-00-4
GC/Hi-Res MS, SIM	EPA 8290A	Total Tetrachlorodibenzofuran (TCDF) CAS#55722-27-5
GC/Hi-Res MS, SIM	EPA 8290A	Total Pentachlorodibenzofuran (PeCDF) CAS#30402-15-4
GC/Hi-Res MS, SIM	EPA 8290A	Total Hexachlorodibenzofuran (HxCDF) CAS#55684-94-1
GC/Hi-Res MS, SIM	EPA 8290A	Total Heptachlorodibenzofuran (HpCDF) CAS#38998-75-3
GC/Hi-Res MS, SIM	EPA 8290A	Total PCDF
GC/Hi-Res MS, SIM	EPA 8290A	Total PCDD

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668A	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668A	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668A	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,2',3,3',5-) (PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668A	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668A	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668A	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668A	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668A	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668A	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668A	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668A	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668A	Decachlorobiphenyl
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#1-Cal/RTW Chlorobiphenyl (2-) (PCB 1)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#2 Chlorobiphenyl (3-) (PCB 2)
GC/Hi-Res MS	EPA 1668C	Cl1-BZ#3-RTW Chlorobiphenyl (4-) (PCB 3)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#4/#10-RTW Dichlorobiphenyl (2,2'-) (PCB 4)/ Dichlorobiphenyl (2,6-) (PCB 10)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#9 Dichlorobiphenyl (2,5-) (PCB 9)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#7 Dichlorobiphenyl (2,4-) (PCB 7)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#6 Dichlorobiphenyl (2,3'-) (PCB 6)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#5 Dichlorobiphenyl (2,3-) (PCB 5)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#8 Dichlorobiphenyl (2,4'-) (PCB 8)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#19-RTW Trichlorobiphenyl (2,2',6-) (PCB 19)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#14 Dichlorobiphenyl (3,5-) (PCB 14)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#30 Trichlorobiphenyl (2,4,6-) (PCB 30)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#18 Trichlorobiphenyl (2,2',5-) (PCB 18)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#11 Dichlorobiphenyl (3,3'-) (PCB 11)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#17 Trichlorobiphenyl (2,2',4-) (PCB 17)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#12 Dichlorobiphenyl (3,4-) (PCB 12)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#27 Trichlorobiphenyl (2,3',6-) (PCB 27)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#13 Dichlorobiphenyl (3,4'-) (PCB 13)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#24 Trichlorobiphenyl (2,3,6-) (PCB 24)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#16 Trichlorobiphenyl (2,2',3-) (PCB 16)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#32 Trichlorobiphenyl (2,4',6-) (PCB 32)
GC/Hi-Res MS	EPA 1668C	Cl2-BZ#15-RTW Dichlorobiphenyl (4,4'-) (PCB 15)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#34 Trichlorobiphenyl (2,3',5'-) (PCB 34)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#23 Trichlorobiphenyl (2,3,5-) (PCB 23)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#54-RTW Tetrachlorobiphenyl (2,2',6,6'-) (PCB 54)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#29-Cal Trichlorobiphenyl (2,4,5-) (PCB 29)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#50-Cal Tetrachlorobiphenyl (2,2',4,6-) (PCB 50)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#26 Trichlorobiphenyl (2,3',5-) (PCB 26)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#25 Trichlorobiphenyl (2,3',4-) (PCB 25)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#53 Tetrachlorobiphenyl (2,2',5,6'-) (PCB 53)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#-31 Trichlorobiphenyl (2,4',5-) (PCB 31)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#28 Trichlorobiphenyl (2,4,4'-) (PCB 28)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#33 Trichlorobiphenyl (2,3',4'-) (PCB 33)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#21/#20 Trichlorobiphenyl (2,3,4-) (PCB 21)/ Trichlorobiphenyl (2,3,3'-) (PCB 20)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#51 Tetrachlorobiphenyl (2,2',4,6'-) (PCB 51)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#45 Tetrachlorobiphenyl (2,2',3,6-) (PCB 45)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#22 Trichlorobiphenyl (2,3,4'-) (PCB 22)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#73/#46 Tetrachlorobiphenyl (2,3',5',6-) (PCB 73)/ Tetrachlorobiphenyl (2,2',3,6'-) (PCB 46)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#69 Tetrachlorobiphenyl (2,3',4,6-) (PCB 69)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#43 Tetrachlorobiphenyl (2,2',3,5-) (PCB 43)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#36 Trichlorobiphenyl (3,3',5-) (PCB 36)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#52 Tetrachlorobiphenyl (2,2',5,5'-) (PCB 52)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#48 Tetrachlorobiphenyl (2,2',4,5-) (PCB 48)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#49 Tetrachlorobiphenyl (2,2',4,5'-) (PCB 49)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#104-RTW Pentachlorobiphenyl (2,2',4,6,6'-) (PCB 104)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#47 Tetrachlorobiphenyl (2,2',4,4'-) (PCB 47)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#65/#75/#62 Tetrachlorobiphenyl (2,3,5,6-) (PCB 65)/ Tetrachlorobiphenyl (2,4,4',6-) (PCB 75)/ Tetrachlorobiphenyl (2,3,4,6-) (PCB 62)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#39 Trichlorobiphenyl (3,4',5-) (PCB 39)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#38 Trichlorobiphenyl (3,4,5-) (PCB 38)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#44 Tetrachlorobiphenyl (2,2',3,5'-) (PCB 44)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#59 Tetrachlorobiphenyl (2,3,3',6-) (PCB 59)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#42 Tetrachlorobiphenyl (2,2',3,4'-) (PCB 42)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#71 Tetrachlorobiphenyl (2,3',4',6-) (PCB 71)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#35 Trichlorobiphenyl (3,3',4-) (PCB 35)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#41 Tetrachlorobiphenyl (2,2',3,4-) (PCB 41)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#72 Tetrachlorobiphenyl (2,3',5,5'-) (PCB 72)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#96 Pentachlorobiphenyl (2,2',3,6,6'-) (PCB 96)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#103 Pentachlorobiphenyl (2,2',4,5',6-) (PCB 103)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#68/#64 Tetrachlorobiphenyl (2,3',4,5'-) (PCB 68) / Tetrachlorobiphenyl (2,3,4',6-) (PCB 64)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#40 Tetrachlorobiphenyl (2,2',3,3'-) (PCB 40)
GC/Hi-Res MS	EPA 1668C	Cl3-BZ#37-RTW Trichlorobiphenyl (3,4,4'-) (PCB 37)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#100 Pentachlorobiphenyl (2,2',4,4',6-) (PCB 100)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#94 Pentachlorobiphenyl (2,2',3,5,6'-) (PCB 94)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#57 Tetrachlorobiphenyl (2,3,3',5-) (PCB 57)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#67/#58 Tetrachlorobiphenyl (2,3',4,5-) (PCB 67)/ Tetrachlorobiphenyl (2,3,3',5'-) (PCB 58)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#102 Pentachlorobiphenyl (2,2',4,5,6'-) (PCB 102)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#61 Tetrachlorobiphenyl (2,3,4,5-) (PCB 61)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#98 Pentachlorobiphenyl (2,2',3,4',6'-) (PCB 98)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#76 Tetrachlorobiphenyl (2,3',4',5'-) (PCB 76)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#93 Pentachlorobiphenyl (2,2',3,5,6-) (PCB 93)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#63 Tetrachlorobiphenyl (2,3,4',5-) (PCB 63)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#121 Pentachlorobiphenyl (2,3',4,5',6-) (PCB 121)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#95/#88 Pentachlorobiphenyl (2,2',3,5',6-) (PCB 95) / Pentachlorobiphenyl (2,2',3,4,6-) (PCB 88)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#74 Tetrachlorobiphenyl (2,4,4',5-) (PCB 74)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#155-RTW Hexachlorobiphenyl (2,2',4,4',6,6'-) (PCB 155)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#70 Tetrachlorobiphenyl (2,3',4',5-) (PCB 70)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#66 Tetrachlorobiphenyl (2,3',4,4'-) (PCB 66)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#91 Pentachlorobiphenyl (2,2',3,4',6-) (PCB 91)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#80 Tetrachlorobiphenyl (3,3',5,5'-) (PCB 80)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#55 Tetrachlorobiphenyl (2,3,3',4-) (PCB 55)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#92 Pentachlorobiphenyl (2,2',3,5,5'-) (PCB 92)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#89/#84 Pentachlorobiphenyl (2,2',3,4,6'-) (PCB 89)/ Pentachlorobiphenyl (2,2',3,3',6-) (PCB 84)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#101/#90 Pentachlorobiphenyl (2,2',4,5,5'-) (PCB 101)/ Pentachlorobiphenyl (2,2',3,4',5-) (PCB 90)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#56 Tetrachlorobiphenyl (2,3,3',4'-) (PCB 56)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#113 Pentachlorobiphenyl (2,3,3',5',6-) (PCB 113)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#99 Pentachlorobiphenyl (2,2',4,4',5-) (PCB 99)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#150 Hexachlorobiphenyl (2,2',3,4',6,6'-) (PCB 150)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#60 Tetrachlorobiphenyl (2,3,4,4'-) (PCB 60)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#152 Hexachlorobiphenyl (2,2',3,5,6,6'-) (PCB 152)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#119 Pentachlorobiphenyl (2,3',4,4',6-) (PCB 119)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#83/#125/#112 Pentachlorobiphenyl (2,2',3,3',5-) (PCB 83)/ Pentachlorobiphenyl (2,3',4',5',6-) (PCB 125) / Pentachlorobiphenyl (2,3,3',5,6-) (PCB 112)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#86/#109 Pentachlorobiphenyl (2,2',3,4,5-) (PCB 86)/ Pentachlorobiphenyl (2,3,3',4,6-) (PCB 109)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#97 Pentachlorobiphenyl (2,2',3,4',5'-) (PCB 97)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#116 Pentachlorobiphenyl (2,3,4,5,6-) (PCB 116)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#87/#111 Pentachlorobiphenyl (2,2',3,4,5'-) (PCB 87) / Pentachlorobiphenyl (2,3,3',5,5'-) (PCB 111)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#145 Hexachlorobiphenyl (2,2',3,4,6,6'-) (PCB 145)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#148 Hexachlorobiphenyl (2,2',3,4',5,6'-) (PCB 148)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#79 Tetrachlorobiphenyl (3,3',4,5'-) (PCB 79)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#154-Cal Hexachlorobiphenyl (2,2',4,4',5,6'-) (PCB 154)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#78 Tetrachlorobiphenyl (3,3',4,5-) (PCB 78)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#136 Hexachlorobiphenyl (2,2',3,3',6,6'-) (PCB 136)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#117 Pentachlorobiphenyl (2,3,4',5,6-) (PCB 117)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#115 Pentachlorobiphenyl (2,3,4,4',6-) (PCB 115)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#85 Pentachlorobiphenyl (2,2',3,4,4'-) (PCB 85)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#120 Pentachlorobiphenyl (2,3',4,5,5'-) (PCB 120)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#110 Pentachlorobiphenyl (2,3,3',4',6-) (PCB 110)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#81 Tetrachlorobiphenyl (3,4,4',5-) (PCB 81)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#151 Hexachlorobiphenyl (2,2',3,5,5',6-) (PCB 151)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#135 Hexachlorobiphenyl (2,2',3,3',5,6'-) (PCB 135)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#82 Pentachlorobiphenyl (2,2',3,3',4-) (PCB 82)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#144 Hexachlorobiphenyl (2,2',3,4,5',6-) (PCB 144)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#147/#149 Hexachlorobiphenyl (2,2',3,4',5,6-) (PCB 147)/ Hexachlorobiphenyl (2,2',3,4',5',6-) (PCB 149)
GC/Hi-Res MS	EPA 1668C	Cl4-BZ#77-RTW Tetrachlorobiphenyl (3,3',4,4'-) (PCB 77)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#143/#139 Hexachlorobiphenyl (2,2',3,4,5,6'-) (PCB 143) / Hexachlorobiphenyl (2,2',3,4,4',6-) (PCB 139)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#124 Pentachlorobiphenyl (2,3',4',5,5'-) (PCB 124)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#108 Pentachlorobiphenyl (2,3,3',4,5'-) (PCB 108)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#107/#123 Pentachlorobiphenyl (2,3,3',4',5-) (PCB 107) / Pentachlorobiphenyl (2,3',4,4',5'-) (PCB 123)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#140 Hexachlorobiphenyl (2,2',3,4,4',6'-) (PCB 140)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#188-Cal/RTW Heptachlorobiphenyl (2,2',3,4',5,6,6'-) (PCB 188)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#134 Hexachlorobiphenyl (2,2',3,3',5,6-) (PCB 134)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#106 Pentachlorobiphenyl (2,3,3',4,5-) (PCB 106)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#133 Hexachlorobiphenyl (2,2',3,3',5,5'-) (PCB 133)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#142 Hexachlorobiphenyl (2,2',3,4,5,6-) (PCB 142)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#118 Pentachlorobiphenyl (2,3',4,4',5-) (PCB 118)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#131 Hexachlorobiphenyl (2,2',3,3',4,6-) (PCB 131)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#184 Heptachlorobiphenyl (2,2',3,4,4',6,6'-) (PCB 184)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#165 Hexachlorobiphenyl (2,3,3',5,5',6-) (PCB 165)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#146 Hexachlorobiphenyl (2,2',3,4',5,5'-) (PCB 146)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#161 Hexachlorobiphenyl (2,3,3',4,5',6-) (PCB 161)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#122 Pentachlorobiphenyl (2,3,3',4',5'-) (PCB 122)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#168 Hexachlorobiphenyl (2,3',4,4',5',6-) (PCB 168)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#114 Pentachlorobiphenyl (2,3,4,4',5-) (PCB 114)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#153 Hexachlorobiphenyl (2,2',4,4',5,5'-) (PCB 153)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#132 Hexachlorobiphenyl (2,2',3,3',4,6'-) (PCB 132)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#179 Heptachlorobiphenyl (2,2',3,3',5,6,6'-) (PCB 179)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#141 Hexachlorobiphenyl (2,2',3,4,5,5'-) (PCB 141)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#176 Heptachlorobiphenyl (2,2',3,3',4,6,6'-) (PCB 176)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#105 Pentachlorobiphenyl (2,3,3',4,4'-) (PCB 105)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#137 Hexachlorobiphenyl (2,2',3,4,4',5-) (PCB 137)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#127 Pentachlorobiphenyl (3,3',4,5,5'-) (PCB 127)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#186 Heptachlorobiphenyl (2,2',3,4,5,6,6'-) (PCB 186)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#130/#164 Hexachlorobiphenyl (2,2',3,3',4,5'-) (PCB 130) / Hexachlorobiphenyl (2,3,3',4',5',6-) (PCB 164)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#178 Heptachlorobiphenyl (2,2',3,3',5,5',6-) (PCB 178)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#138 Hexachlorobiphenyl (2,2',3,4,4',5'-) (PCB 138)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#163/#160 Hexachlorobiphenyl (2,3,3',4',5,6-) (PCB 163) / Hexachlorobiphenyl (2,3,3',4,5,6-) (PCB 160)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#129/#158 Hexachlorobiphenyl (2,2',3,3',4,5-) (PCB 129)/ Hexachlorobiphenyl (2,3,3',4,4',6-) (PCB 158)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#182/#175 Heptachlorobiphenyl (2,2',3,4,4',5,6'-) (PCB 182)/ Heptachlorobiphenyl (2,2',3,3',4,5',6-) (PCB 175)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#187 Heptachlorobiphenyl (2,2',3,4',5,5',6-) (PCB 187)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#183 Heptachlorobiphenyl (2,2',3,4,4',5',6-) (PCB 183)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#166 Hexachlorobiphenyl (2,3,4,4',5,6-) (PCB 166)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#159 Hexachlorobiphenyl (2,3,3',4,5,5'-) (PCB 159)
GC/Hi-Res MS	EPA 1668C	Cl5-BZ#126-RTW Pentachlorobiphenyl (3,3',4,4',5-) (PCB 126)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#185 Heptachlorobiphenyl (2,2',3,4,5,5',6-) (PCB 185)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#162 Hexachlorobiphenyl (2,3,3',4',5,5'-) (PCB 162)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#174 Heptachlorobiphenyl (2,2',3,3',4,5,6'-) (PCB 174)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#128 Hexachlorobiphenyl (2,2',3,3',4,4'-) (PCB 128)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#167 Hexachlorobiphenyl (2,3',4,4',5,5'-) (PCB 167)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#202-RTW Octachlorobiphenyl (2,2',3,3',5,5',6,6'-) (PCB 202)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#181 Heptachlorobiphenyl (2,2',3,4,4',5,6-) (PCB 181)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#177 Heptachlorobiphenyl (2,2',3,3',4,5',6'-) (PCB 177)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#204/#200-Cal Octachlorobiphenyl (2,2',3,4,4',5,6,6'-) (PCB 204) / Octachlorobiphenyl (2,2',3,3',4,5,6,6'-) (PCB 200)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#171 Heptachlorobiphenyl (2,2',3,3',4,4',6-) (PCB 171)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#173 Heptachlorobiphenyl (2,2',3,3',4,5,6-) (PCB 173)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#172 Heptachlorobiphenyl (2,2',3,3',4,5,5'-) (PCB 172)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#192 Heptachlorobiphenyl (2,3,3',4,5,5',6-) (PCB 192)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#156 Hexachlorobiphenyl (2,3,3',4,4',5-) (PCB 156)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#157 Hexachlorobiphenyl (2,3,3',4,4',5'-) (PCB 157)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#180 Heptachlorobiphenyl (2,2',3,4,4',5,5'-) (PCB 180)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#193 Heptachlorobiphenyl (2,3,3',4',5,5',6-) (PCB 193)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#197 Octachlorobiphenyl (2,2',3,3',4,4',6,6'-) (PCB 197)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#191 Heptachlorobiphenyl (2,3,3',4,4',5',6-) (PCB 191)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#199 Octachlorobiphenyl (2,2',3,3',4,5,5',6'-) (PCB 199)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#198 Octachlorobiphenyl (2,2',3,3',4,5,5',6-) (PCB 198)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#201 Octachlorobiphenyl (2,2',3,3',4,5',6,6'-) (PCB 201)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#170 Heptachlorobiphenyl (2,2',3,3',4,4',5-) (PCB 170)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#190 Heptachlorobiphenyl (2,3,3',4,4',5,6-) (PCB 190)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#196 Octachlorobiphenyl (2,2',3,3',4,4',5,6'-) (PCB 196)

Biological Tissue		
Technology	**Method**	**Analyte**
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#203 Octachlorobiphenyl (2,2',3,4,4',5,5',6-) (PCB 203)
GC/Hi-Res MS	EPA 1668C	Cl6-BZ#169-RTW Hexachlorobiphenyl (3,3',4,4',5,5'-) (PCB 169)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#208-RTW Nonachlorobiphenyl (2,2',3,3',4,5,5',6,6'-) (PCB 208)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#207 Nonachlorobiphenyl (2,2',3,3',4,4',5,6,6'-) (PCB 207)
GC/Hi-Res MS	EPA 1668C	Cl7-BZ#189-RTW Heptachlorobiphenyl (2,3,3',4,4',5,5'-) (PCB 189)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#195 Octachlorobiphenyl (2,2',3,3',4,4',5,6-) (PCB 195)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#194 Octachlorobiphenyl (2,2',3,3',4,4',5,5'-) (PCB 194)
GC/Hi-Res MS	EPA 1668C	Cl8-BZ#205-RTW Octachlorobiphenyl (2,3,3',4,4',5,5',6-) (PCB 205)
GC/Hi-Res MS	EPA 1668C	Cl9-BZ#206-Cal/RTW Nonachlorobiphenyl (2,2',3,3',4,4',5,5',6-) (PCB 206)
GC/Hi-Res MS	EPA 1668C	Cl10-BZ#209-Cal/RTW Decachlorobiphenyl (PCB 209)
GC/Hi-Res MS	EPA 1668C	Monochlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Dichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Trichlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Tetrachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Pentachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Hexachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Heptachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Octachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Nonachlorobiphenyls
GC/Hi-Res MS	EPA 1668C	Decachlorobiphenyl
Preparation	**Method**	**Type**
Extraction	EPA 3570	Microscale Extraction (MSE)
Extraction	Alpha SOP ID 2264	Tissue Extraction
Cleanup	EPA 3630C	Silica Gel Cleanup
Cleanup	EPA 3660B	Sulfur Removal Cleanup
Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Air and Emissions		
Technology	**Method**	**Analyte**
GC/TCD/FID	EPA Method 3C	Carbon Dioxide
GC/TCD/FID	EPA Method 3C	Nitrogen
GC/TCD/FID	EPA Method 3C	Oxygen
GC/TCD/FID	EPA Method 3C	Methane
GC/FID	EPA TO-12	Non-Methane Organic Compounds
GC/MS	EPA TO-15	1,1,1,2-tetrachloroethane
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-dichloroethene
GC/MS	EPA TO-15	1,1-dichloropropene
GC/MS	EPA TO-15	1,2,3-trichlorobenzene
GC/MS	EPA TO-15	1,2,3-trichloropropane
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-dibromo-3-chloropropane
GC/MS	EPA TO-15	1,2-dibromoethane
GC/MS	EPA TO-15	1,2-dichlorobenzene
GC/MS	EPA TO-15	1,2-dichloroethane
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	1,3-dichloropropane
GC/MS	EPA TO-15	1,4-dichlorobenzene
GC/MS	EPA TO-15	1,4-dioxane
GC/MS	EPA TO-15	2,2,4-trimethylpentane
GC/MS	EPA TO-15	2,2-dichloropropane
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	3-chloropropene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	4-chlorotoluene
GC/MS	EPA TO-15	4-ethyl toluene
GC/MS	EPA TO-15	4-methyl-2-pentanone (MIBK)
GC/MS	EPA TO-15	acetone
GC/MS	EPA TO-15	acetonitrile
GC/MS	EPA TO-15	acrolein
GC/MS	EPA TO-15	acrylonitrile
GC/MS	EPA TO-15	benzene
GC/MS	EPA TO-15	benzyl chloride
GC/MS	EPA TO-15	bromobenzene
GC/MS	EPA TO-15	bromodichloromethane
GC/MS	EPA TO-15	bromoform
GC/MS	EPA TO-15	bromomethane
GC/MS	EPA TO-15	carbon disulfide
GC/MS	EPA TO-15	carbon tetrachloride
GC/MS	EPA TO-15	chlorobenzene
GC/MS	EPA TO-15	chlorodifluoromethane
GC/MS	EPA TO-15	chloroethane
GC/MS	EPA TO-15	chloroform
GC/MS	EPA TO-15	chloromethane
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	dibromomethane
GC/MS	EPA TO-15	dichlorodifluoromethane
GC/MS	EPA TO-15	dichlorofluoromethane
GC/MS	EPA TO-15	diisopropyl ether
GC/MS	EPA TO-15	ethanol
GC/MS	EPA TO-15	ethyl acetate
GC/MS	EPA TO-15	ethyl ether
GC/MS	EPA TO-15	ethylbenzene
GC/MS	EPA TO-15	Freon 113
GC/MS	EPA TO-15	Freon-114
GC/MS	EPA TO-15	n-heptane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	hexachlorobutadiene
GC/MS	EPA TO-15	hexane
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	methanol
GC/MS	EPA TO-15	methylene chloride
GC/MS	EPA TO-15	methyl methacrylate
GC/MS	EPA TO-15	MTBE
GC/MS	EPA TO-15	naphthalene
GC/MS	EPA TO-15	n-butylbenzene
GC/MS	EPA TO-15	n-propylbenzene
GC/MS	EPA TO-15	octane
GC/MS	EPA TO-15	o-xylene
GC/MS	EPA TO-15	n-pentane
GC/MS	EPA TO-15	p-isopropyltoluene
GC/MS	EPA TO-15	propane
GC/MS	EPA TO-15	propylene
GC/MS	EPA TO-15	sec-butylbenzene
GC/MS	EPA TO-15	styrene
GC/MS	EPA TO-15	tert-amyl methyl ether
GC/MS	EPA TO-15	tert-butlybenzene
GC/MS	EPA TO-15	tert-butyl ethyl ether
GC/MS	EPA TO-15	tetrachloroethene
GC/MS	EPA TO-15	tetrahydrofuran
GC/MS	EPA TO-15	toluene
GC/MS	EPA TO-15	trans-1,2-dichloroethene
GC/MS	EPA TO-15	trans-1,3-dichloropropene
GC/MS	EPA TO-15	trichloroethene
GC/MS	EPA TO-15	trichlorofluoromethane
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	decane
GC/MS	EPA TO-15	undecane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	butane
GC/MS	EPA TO-15	nonane
GC/MS	EPA TO-15	tert butyl alcohol
GC/MS	EPA TO-15	dodecane
GC/MS	EPA TO-15	butyl acetate
GC/MS	EPA TO-15	3-methylthiophene
GC/MS	EPA TO-15	2-ethylthiophene
GC/MS	EPA TO-15	2-methylthiophene
GC/MS	EPA TO-15	thiophene
GC/MS	EPA TO-15	benzothiophene
GC/MS	EPA TO-15	1,2,3-trimethylbenzene
GC/MS	EPA TO-15	indene
GC/MS	EPA TO-15	1,2,4,5-tetramethylbenzene
GC/MS	EPA TO-15	indan
GC/MS	EPA TO-15	1-methylnaphthalene
GC/MS	EPA TO-15	2-methylnaphthalene
GC/MS	EPA TO-15	acetaldehyde
GC/MS	EPA TO-15	Total Xylenes
GC/MS-SIM	EPA TO-15 SIM	1,1,1-Trichloroethane
GC/MS-SIM	EPA TO-15 SIM	1,1,2,2-Tetrachloroethane
GC/MS-SIM	EPA TO-15 SIM	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS-SIM	EPA TO-15 SIM	1,1,2-Trichloroethane
GC/MS-SIM	EPA TO-15 SIM	1,1-Dichloroethane
GC/MS-SIM	EPA TO-15 SIM	1,1-Dichloroethylene
GC/MS-SIM	EPA TO-15 SIM	1,2,4-Trichlorobenzene
GC/MS-SIM	EPA TO-15 SIM	1,2,4-Trimethylbenzene
GC/MS-SIM	EPA TO-15 SIM	1,2-Dibromoethane (EDB, Ethylene dibromide)
GC/MS-SIM	EPA TO-15 SIM	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS-SIM	EPA TO-15 SIM	1,2-Dichloroethane (Ethylene dichloride)
GC/MS-SIM	EPA TO-15 SIM	1,2-Dichloropropane
GC/MS-SIM	EPA TO-15 SIM	1,3,5-Trimethylbenzene
GC/MS-SIM	EPA TO-15 SIM	1,3-Butadiene
GC/MS-SIM	EPA TO-15 SIM	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS-SIM	EPA TO-15 SIM	1,4-Dioxane (1,4- Diethyleneoxide)
GC/MS-SIM	EPA TO-15 SIM	2-Butanone (Methyl ethyl ketone, MEK)

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS-SIM	EPA TO-15 SIM	4-Methyl-2-pentanone (MIBK)
GC/MS-SIM	EPA TO-15 SIM	Acetone
GC/MS-SIM	EPA TO-15 SIM	Acrylonitrile
GC/MS-SIM	EPA TO-15 SIM	Benzene
GC/MS-SIM	EPA TO-15 SIM	Bromodichloromethane
GC/MS-SIM	EPA TO-15 SIM	Bromoform
GC/MS-SIM	EPA TO-15 SIM	Carbon tetrachloride
GC/MS-SIM	EPA TO-15 SIM	Chlorobenzene
GC/MS-SIM	EPA TO-15 SIM	Chlorodibromomethane
GC/MS-SIM	EPA TO-15 SIM	Chloroethane (Ethyl chloride)
GC/MS-SIM	EPA TO-15 SIM	Chloroform
GC/MS-SIM	EPA TO-15 SIM	cis-1,2-Dichloroethylene
GC/MS-SIM	EPA TO-15 SIM	cis-1,3-Dichloropropene
GC/MS-SIM	EPA TO-15 SIM	Dichlorodifluoromethane (Freon-12)
GC/MS-SIM	EPA TO-15 SIM	Dichlorotetrafluoroethane
GC/MS-SIM	EPA TO-15 SIM	Ethylbenzene
GC/MS-SIM	EPA TO-15 SIM	Hexachlorobutadiene
GC/MS-SIM	EPA TO-15 SIM	Isopropylbenzene
GC/MS-SIM	EPA TO-15 SIM	Methyl bromide (Bromomethane)
GC/MS-SIM	EPA TO-15 SIM	Methyl chloride (Chloromethane)
GC/MS-SIM	EPA TO-15 SIM	Methyl tert-butyl ether (MTBE)
GC/MS-SIM	EPA TO-15 SIM	Methylene chloride (Dichloromethane)
GC/MS-SIM	EPA TO-15 SIM	Naphthalene
GC/MS-SIM	EPA TO-15 SIM	n-Butylbenzene
GC/MS-SIM	EPA TO-15 SIM	o-Xylene
GC/MS-SIM	EPA TO-15 SIM	p-Xylene
GC/MS-SIM	EPA TO-15 SIM	sec-Butylbenzene
GC/MS-SIM	EPA TO-15 SIM	Styrene
GC/MS-SIM	EPA TO-15 SIM	Tetrachloroethylene (Perchloroethylene)
GC/MS-SIM	EPA TO-15 SIM	Toluene
GC/MS-SIM	EPA TO-15 SIM	trans-1,2-Dichloroethylene
GC/MS-SIM	EPA TO-15 SIM	trans-1,3-Dichloropropylene
GC/MS-SIM	EPA TO-15 SIM	Trichloroethene (Trichloroethylene)
GC/MS-SIM	EPA TO-15 SIM	Vinyl chloride (Chloroethane)
GC/MS-SIM	EPA TO-15 SIM	Total Xylenes

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2474
2. Not compliant with QSM 5.3 Table B-15

R. Douglas Leonard Jr., VP, PILR SBU

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument, other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	minor changes rewording of sect. 7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practices and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 4 - added list of west streams generated and location of sub #'s. Clarified RT window studies. Added reference to MI Sop. Removed Grand Mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSID Batch requirement can not be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10
12 (LAD 05/11/11)	Removed Tekmar 2000 and 2016 throughout. Sect. 7.3.1 - Removed ~~5890~~ 5970 GC/MS instrument type. Sect. 7.4.7 - Added RRT information. Sect. 8.1 - Added S.C. marginal exceedence criteria. Sect. 9 - Added MDL, LOD and LOQ criteria. Updated Figures	LAD	05/11	05/11
13	Sect. 5 - Changed Cal Mix and ICV Std. Exp. from 7 to 14 days. Sect. 6 - Add Sample preservation info. Sect. 7.4.4.1 - Added S.C. exemption from 2nd order Cal. Sect. 7.5.1 - Added Ex Res mix to LCS. Sect. 7.6.2 - Clarified nothing why samples need to be reanalyzed. Sect. 8.1 - Added 10% rule for LCS, ICV and MS/D. Sect. 9 - Added LOD/LOQ definitions, Table 1 - Reworded CA for ICV.	LAD	03/12	03/12
14	Sect. 1 and 7 - Removed Quikform references and added reporting from KIMS. Sect. 7 - Removed Soil 200 ug/kg level and added 80.% level. Sect. 8 - Added additional marginal exceedance information. Throughout - Fixed typos and made minor edits.	LAD	04/13	04/13
15	Sect. 4 - Removed 5890, 5972 and Tekmar references. (5970 too). Sect. 10 - Updated and added references. Table 3 - Added DoD QSM Ver. 5.0 QC requirements. Renumbered Tables 3, 4, 5.	LAD	04/14	04/14

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
16	Sect. 1 – Added sand to m. blk def. Sect. 5 – Separated cal std into VOA gases and non-gases, clarified VOA gases exp. date, combined Surr & IS std, added sand. Sect. 6 – Added Acrolein and Acrylonitrile preservation. Sect. 7 – Added poor purgers % RSD calibration info, added CCV criteria, added use of sand in soil Blank and LCS's, updated cal spike prep. Sect 8 – Added additional LCS criteria. Updated Figures 1 – 3.	LAD	07/16	07/16
17	Sect. 7 - Update BFB Method naming convention, Update Soil calibration levels, Added % Error calculation. Sect. 9. Added LLOQ reference to LOQ verification.	LAD	03/17	03/17
18	Sect 1, 8, 9 and/or Table 1 - Added LLOQ definition, and LLOQ verification criteria, clarified PQL, LOQ and LLOQ. Added % Error criteria Sect. 7 - Corrected RSE requirement	LAD	06/17	06/17
19	Sect. 5 - Changed IS and Surrogate Standard expiration date from 14 to 30 days. Removed references for 1-Chlorohexane. Removed Table 2 - DoD/Isom 4.2 QC criteria. Updated logbook example. Updated references	LAD	10/18	10/18
20	Sect. 4 - Updated columns, data acquisition and processing systems. Sect. 5 - Updated final concentrations and expiration dates for non-gas stds. Sect. 7 - Updated calib. high pt. conc.	LAD	01/20	01/20
21	Sect. 5.3.1 - Added requirement that ampulated standards must not be used after the expiration of the manufacturer expiration date.	LAD	06/20	06/20
22	Sect 1 & 7.6.2 - Changed 5g of baked sand to 5mL DI H2O for LL Soil blanks. Sect. 7.4.2 - Changed CCC criteria from ≤ 30% to ≤ 20%. Sect. 7.6.3 - Changed 40mL methanol to 20 mL to 800mL methanol to 40mL. Sect. 4.2 - added MD 5975.	LAD	06/21	06/21

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

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TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

PRACTICAL QUANTITATION LIMIT (PQL), LIMIT OF QUANTITATION (LOQ) AND LOWER LIMIT OF QUANTITATION (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices

common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, deionized water equal to the amount of preservative. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil

samples must be decanted, and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically, 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire concentrator must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph (GC): Hewlett Packard 6890.

4.2 Mass Spectrometer (MS): HP5973 or HP5975

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Column: RTX-VMS, 30 meter, 0.25 mm ID or equivalent.

4.5 Purge and Trap: Archon or Centurion auto samplers, and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Agilent Chemstation or equivalent.

4.9 Data System: KIMS is used for processing data. The Target software is used for generating forms.

5.0 REAGENTS AND STANDARDS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”. After ampulated standards are cranked open, the standard is transferred to a screw top vial and stored in a freezer.

5.3.1 Ampulated standards must not be used past the manufacturers expiration date.

The expiration date for all standards except volatile gases is six months from date of opening the ampule.

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix (without gases) – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

1,2-Dibromo-3-chloropropane
1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Butanone
2-Chloroethylvinyl ether
2-Chlorotoluene
2-Hexanone
4-Chlorotoluene
4-Methyl-2-pentanone
Acetone
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Carbon disulfide
Carbon Tetrachloride
Chlorobenzene
Chloroform
cis-1,2-Dichloroethene
cis-1,3-Dichloropropene
Cyclohexane
Dibromochloromethane
Dibromomethane
Ethylbenzene
Hexachlorobutadiene
Idomethane
Isopropylbenzene
Methyl tert-butyl ether
Methylene chloride
Naphthalene
n-Butylbenzene
n-Propylbenzene
p-Isopropyltoluene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethene
Tetrahydrofuran
Toluene
trans-1,2-Dichloroethene
trans-1,3-Dichloropropene
Trichloroethene
Vinyl Acetate

5.3.2.2 Gases Calibration Mix - Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Bromomethane
Chloromethane
Dichlorodifluoromethane
Trichlorofluoromethane
Vinyl Chloride
Chloroethane

5.3.2.3 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl chloride	Methyl acetate
Chloroprene	Methyl methacrylate
Diethyl ether	Methyl tert-butyl ether
cis-1,4-Dichloro-2-butene	Pentachloroethane (only by special request)
trans-1,4-Dichloro-2-butene	Propionitrile
1,4-Dioxane	Tertiary-amyl methyl ether
di-Isopropyl Ether	Tertiary-butyl alcohol
Ethyl methacrylate	1,3,5-Trichlorobenzene
Ethyl tertiary-butyl ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.4 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

5.3.2.5 Surrogate/Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 30 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Internal Standards	Surrogate Standards
Pentafluorobenzene	4-Bromofluorobenzene
1,4-Difluorobenzene	1,2-Dichloroethane-D_4
Chlorobenzene-D_5	Toluene-D_8
1,4-Dichlorobenzene-D_4	Dibromofluoromethane

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 50 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

5.4 Organic Free Sand – Ottawa Sand or equivalent baked at 110 ºC overnight

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples

All aqueous samples are collected in 40 mL VOA bottles with no headspace, preserved with 1:1 HCl to a pH of <2 and stored at <6˚C until analysis. Aqueous samples must be analyzed within 14 days from sample collection if preserved and within 7 days from sample collection if unpreserved.

Samples requiring Acrolein and Acrylonitrile analysis, require preservation of pH of 4-5 and cool to 0-6ºC.

6.2 Soil Samples

Soil samples arriving at the laboratory in Terra-core or Encores Soil samplers must be extruded into water or sodium bisulfate within 48 hours of sampling. Soils samples extruded into water must be frozen at -15˚C ± 5˚C until analysis. Soil sample extruded into sodium bisulfate must be stored at <6 ˚C until analysis.

Medium level soil (methanol preserved) samples are sampled into pre-weighed vials containing 5 mLs methanol. Methanol preserved soil samples must be stored at <6˚C from the time of receipt at the lab until analysis.

Bulk soil samples are stored at <6 ˚C until analysis.

All soil/sediments must be analyzed within 14 days from sample collection.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS - Conventions for all instruments are as follows:

- Sub-Directory for data acquisition: C:\HPCHEM\1\DATA
- Tune file: BFB.U
- Method files:

  For BFB Tune: VOABFBAQ.M (waters) or VOABFBSL.M (soils)
  For all samples and standards: I8A05(xx)D.M
  where: I = instrument ID (Each instrument is given a unique identifier).
  A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

XX = the calibration number in chronological order

- Data files:
  For BFB: IB_ _ _.D

  where: I is the instrument ID
  _ _ _ is a number in chronological order from 000 to 999.

  For all other data files: I_ _ _ _.D
  Where: I is the instrument ID
  _ _ _ _ is a number in chronological order from 0000 to 9999.

  This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

Column:		RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:		150°
Run time:		8 minutes
Scan start time:		3 minutes
Scan parameters:		not to exceed 2 sec per scan
Mass range:		35-300
Number of A/D samples:		8
GC peak threshold:		1000 counts
Threshold:		10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All

calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

Purge and Trap conditions:

Parameter	Aqueous	Soil
Standby:	35°	35°
Prepurge:	0 min	0 min
Preheat Temp:	Ambient	40°
Sample Temp:	Ambient	40°
Purge:	11 min	11 min
Purge Flow Rate	~24-40 mL/min	~24-40 mL/min
Dry purge:	2-4 min	2-4 min
Desorb preheat:	245°	245°
Desorb Temp:	250°	250°
Desorb Flow Rate:	~15 mL/min	~15 mL/min
Desorb time:	2-5 min	2-5 min
Dry purge:	2-4 min	2-4 min
Bake Time:	10 min	10 min
Bake Temp:	260°	260°
Auto drain:	On	On
Bake gas by pass:	Off	Off
Valve Temp:	120°	120°
Line Temp:	120°	120°
Runs per sample:	1	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

Please refer to the Encon, Archon and Centurion Opperating manuals for more specifics on programming features.

7.4.1 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations: 1.0, 5.0, 20, 50, 100 and 150 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 150 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water to generate a 6-point initial calibration curve:

Notes	STD. ID	CAL. Mix 100 ug/mL	Extras Mix 100 ug/mL
AQ curve only	VSTD001	1 uL	1 uL
	VSTD005	5 uL	5 uL
SL curve only	VSTD010	10 uL	10 uL
	VSTD020	20 uL	20 uL
CCV	VSTD050	50 uL	50 uL
	VSTD100	100 uL	100 uL
	VSTD150	150 uL	150 uL

The Surrogate & Internal Standard is spiked by the autosampler. The Archon Surrogate/IS Mix is at 250 ug/ml and the instrument spikes 1 ul. The Centurion Surrogate/IS Mix is prepared at 50 ug/ml and the instrument spikes 5 ul.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.2 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 20%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.2.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. For the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Note 1: For poor purging compounds like acetone, the %RSD value may exceed the method acceptance limit of 15% but meet the acceptance criteria for the linear and quadratic calibration models. The average calibration model should still be used because this calibration model is more accurate at concentrations near the LOQ than either the linear or quadratic calibration models.

This is common for acetone but also may apply to other poor purging ketones.

In any instance the % RSD must be below 30%.

Note 2: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.4.2.2 Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\ Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤15% for good performing compounds and ≤30% for poor performing compounds.

7.4.3 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards, and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

For projects or clients requiring DoD QSM, current revision, all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.4 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.4, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM, current version, all project analytes must have $\pm$ 20%D.

For all other projects, all project analytes should have $\pm$ 30%D ($\pm$ 40%D for poor performers).

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.5 Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS and Extras standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution plus 5 g baked Ottawa sand, in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

NOTE: If the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water, spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the 1/10th the measured amount in any sample or 1/10th the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

For projects or clients requiring DoD QSM, current version, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner like the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and

field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

NOTE: If sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Archon Autosamplers

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

7.6.1.2 Centurion Autosamplers

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted, or duplicated, it must be noted in the comments section of the injection logbook. Additional information may be needed to assure that any questions that arise during the review process can be answered.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor samples analyzed after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil

logbook. If possible, the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the autosampler or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. In this case, the Archon units may be used for the preparative step.

7.6.3 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/-

0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed, and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 800 uL of the extract into 40 mL of organic-free laboratory reagent grade water.
IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating

the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into Kims. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criterion does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client, and project specific Data Quality Objectives and on review of chromatograms. The Department Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank – Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.

8.2 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. For non-DOD clients, the exceedances from the laboratory established limits or nominal limits must be less than ten percent of the client compound list. For DOD clients, all the compounds of interest must fall within either Katahdin's statistically derived limits or the DOD QSM, current version, limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state. Additionally, the laboratory statistically derived LCS limits should fall within 70-130%.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

For projects or clients requiring DoD QSM, current version, all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.3 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.4 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be

evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

8.5 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260C.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, current Version.

The 2009 TNI Standards

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all a nalytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also. % Error ≤ 30%	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances.	If the surrogate recoveries in the ICV are low but the target analytes are acceptable, narrate. If the ICV recovery is high but the sample results are <PQL, narrate. If the ICV is out but the batch LCS is in criteria, narrate.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL/LLOQ with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
MDL Studies, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-21	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Di-Isopropyl ether	45	43, 87
Ethylbezene	91	106
Ethyl methacrylate	69	41, 99
Ethyl tertiary-butyl ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl alcohol	43	41, 42
Isopropylbezene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene chloride	84	86, 49
Methyl acetate	43	74
Methyl methacrylate	69	41, 100
4-Methyl-2-pentanone	43	58, 85, 100
Methyl tert-butyl ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
n-Propylbenzene	91	120
Styrene	104	78
Tertiary-amyl methyl ether	73	55, 87, 71
Tertiary-butyl alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
Xylenes (Total)	106	91

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 5

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenzene	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butyl ether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl ether			n-Butylbenzene
Carbon disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES

GCMS-T INSTRUMENT RUNLOG **DATE/TIME OF BFB INJECTION:**

Method - Check One:	8260	8260 - 10 mL	8260 SIM	624	524
STD IDs: * BFB STD:	CAL STD:	GAS STD:	XTRAs STD:	LCS/MS STD:	
SURR STD:	IS STD:	Prep Method	Criteria	pH Paper Lot #:	KI Paper Lot #

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	5030	5035	1311	KAS	DoD	QAPP	Y/N	ANALYST	pH	TRC	COMMENTS

* Refer to page 1 for standard preparation instructions.

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0000007

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS VOLATILES LABORATORY

REVIEWED BY/DATE:

AMP4638 Rev. PVOC/4RO Mix RLC
Ultra Scientific 6/2/16
4ST-100-1 JAP
Lot CP-1618 exp 5/31/19

AMP 4639 REVISED PVOC/4RO MIX 060616 AAB
SUPELCO 47578-U
LOT XA13795V X 04/2018

Amp 4640 ↓ 41

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-502A-R2-10X
VOC Liquid Mixture - Modified
2.0 mg/mL in Methanol
Lot: 215031197-01
Exp: Mar 24, 2018
1 mL
53 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H336 H320 H315 H311 H332 H301 H350 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Refrig (0-5 °C)
Danger
RLC 6/14/16 EPL

Amp 4642 ↓ 43

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
APP-9-048-R1-20X
Chloroprene
2.0 mg/mL in MeOH
Lot: 216031093
Exp: Mar 04, 2018
1 mL
1 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H336 H320 H315 H311 H332 H301 H351 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Freeze (<-10 °C)
Danger
Rec 6/23/16

Amp 4644 ↓ 45

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-8240C-R3-10X
Appendix IX Volatiles Mix
Varied conc. in MeOH
Lot: 214041128-01
Exp: May 18, 2018
1 mL
12 comp(s)
HIGHLY FLAMMABLE
FOR LABORATOR
H225 H336 H320 H
H311 H332 H301 H
P338 P360 P331 P
P262 P202 P264 P
Storage: Refri
Danger

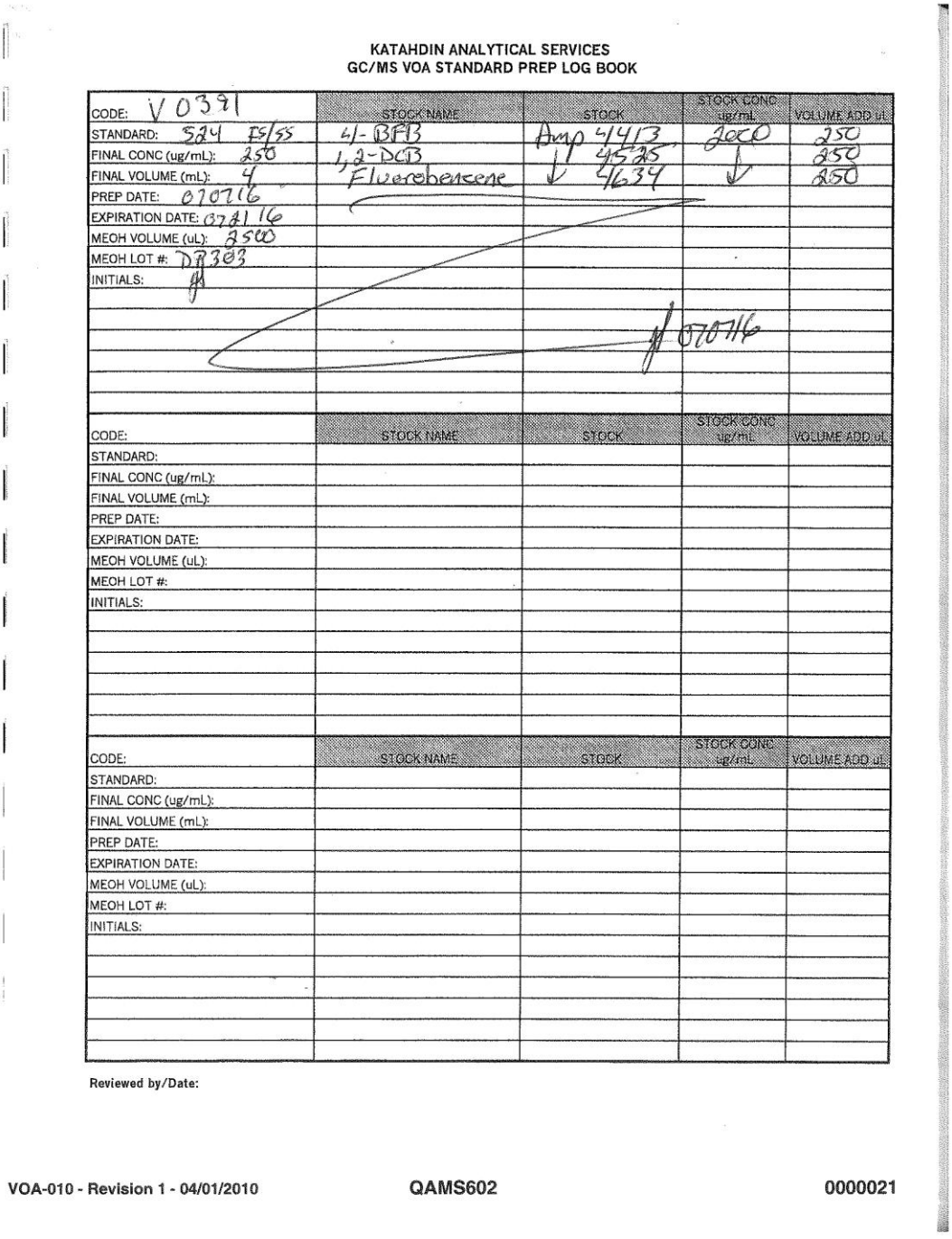
Amp 4646 ↓ 47 ↓ 48

AccuStandard® 125 Market Street • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-502B-10X
Volatile Organic Cmpds - Gases
2.0 mg/mL in MeOH
Lot: 216031159
Exp: Mar 11, 2019
1 mL
6 comp(s)
HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
H225 H330 H320 H315 H311 H332 H301 H350 P338 P360 P331 P404 P262 P202 P264 P284
Storage: Refrig (0-5 °C)
Danger

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V0391	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 524 ES/ES	4-BFB	Amp 4413	2000	250
FINAL CONC (ug/mL): 250	1,2-DCB	4525	↓	250
FINAL VOLUME (mL): 4	Fluorobenzene	↓ 4634		250
PREP DATE: 070716				
EXPIRATION DATE: 073116				
MEOH VOLUME (uL): 3500				
MEOH LOT #: DR303				
INITIALS:				
			070716	

CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS602 0000021

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 020101

Operations Manager: [signature] Date: 1/31/01

QA Officer: [signature] Date: 1.31.01

General Manager: [signature] Date: 2/9/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10
09	Sect. 7.4 - Added additional tune information. Sect. 7.6 - Added 100ul minimum extract vol. & 1ul IS is added for each 100ul aliquot. Sect. 7.5.4 - Added RRT information. Sect. 9.0 - Added MDL, LOD and LOQ information. Table 4 - Added 1,4-Dioxane-d6 Surrogate	LAD	05/11	05/11
10	Sect. 7 - Changed sample volume from 1ul to 2ul. Sect. 8 - Added 10% rule for non-DoD clients. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and updated references. Updated Figure 1. Added Addendum 1 - low level 1,4-Dioxane analysis	LAD	05/12	05/12
11	Sect. 1 and 7 - Removed Quickform reporting and added KIMS. Sect. 8 and Table 1 - Added the Surrogate 1,4-Dioxane-d8. Throughout - Fixed typos and made minor changes.	LAD	03/13	03/13
12	Sect. 4 - Updated instrument and column models. Sect. 7 - updated calibration levels and prep. Sect. 8 - Added marginal exceedance criteria. Updated MS/MSD acceptance criteria. Tables - Added DoD QSM 5.0 QC Requirements. Updated Fig. 2 & 3	LAD	04/14	04/14
13	Sect. 5 - Added Standards to title. Sect. 7 & Appendix 1 - Updated GC/MS operating conditions. Appendix 1. Corrected 1,4 dioxane primary and secondary ions. Add 1,4 dioxane-d4 ions. Changed KAS INC to KAS throughout	LAD	03/16	03/16

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 10-Updated method references. Sect 7 -Added percent error calculation and relative std error calculation. Sect 1.1-updated definitions. Sect. 9.0 - Update. Sect. 7-changed the initial cal. std concentrations. Added compounds to Tables 5 & 7.		09/17	09/17
15	Sect. 7 – updated SSTD3.0 preparation, Changed CCV to SSTD2.0 and conc. to 2.0 ng/ul. Updated runlog example.		01/19	01/19
16	Sect, 5.3.2.6 – Changed IND conc. From 2 to 5 ug/ml. Sect. 7.2- Clarified the tune method file name. Sect 7.4 &Table 1 – Removed tailing factor criteria. Sect. 7.7.4 – Removed QuickForms and added KIMS. Sect. 7.9 – Removed Dept. Manager authorization for maintenance.		07/21	07/21

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-15, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process. Refer to section 8 for Method Blank acceptance criteria

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term LOQ must be used for DoD work. Refer to section 9 for specific LOQ/LLOQ verification requirements

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation of Aqueous Samples for Extractable Semivolatile Analyses”, SOP CA-512, “Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation of

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890.

4.2 Mass Spectrometers (MS): HP5975 or HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: RTX5 SIL MS - 30m, 0.25mm I.D., 25um film thickness, columns (Restek) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

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5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will “boost” the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 4. The final concentration of each compound is 5 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

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7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument is given a unique identifier)
Tune method file: DFTPPXXX.M (XXX chronological order)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (Each instrument is given a unique identifier). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (Each instrument is given a unique identifier).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using “Edit Sample Log Table”. For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each 12 hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

The DFTPP tuning standard can also be used to assess the column performance and injection port inertness by calculating the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should also be present at their normal responses, with no evidence of peak tailing.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 2.0, 7.0, 10.0 and 15.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds need to be calibrated at higher concentrations. A second standard (Mix B) is prepared containing these compounds. The two standards are combined as in the example below. The full aliquot is used and spiked with the appropriate amount of IS.

Example –

Calibration Mix A is prepared containing ALL analytes at 20 ng/ul.

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Calibration Mix B is prepared containing only phenols and phthalates at 20 ng/ul.

For the low standard, 10 ul of mix A and 40 ul of mix B are combined and diluted to 1000 uL with MeCL2. Internal standards are then added prior to analysis.

Calibration Level	Cal-Mix A (All Analytes) Added (uL)	Cal-Mix B (Phenols and Phthalates) Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)	Final Conc. Everything but Phenols and Phthalates (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)
SSTD 0.2	10	40	950	1000	0.20	1.0
SSTD 0.50	25	75	900	1000	0.50	2.0
SSTD 2.0	40	50	310	400	2.0	4.5
SSTD 7.0	70	NA	130	200	7.0	7.0
SSTD 10	100	NA	100	200	10	10
SSTD 15	150	NA	50	200	15	15

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 3.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method file LSPSIMXX.M
After analysis of the six calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

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$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

Acceptance criteria independent of calibration model

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Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.
Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$\text{RSE}=100\ \text{X}\ \sqrt{\sum_{i=1}^{n}\left|\frac{(x'i - xi)^2}{xi}\right|/(\text{n-p})}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must

be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD2.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 2.0 ng/uL.

After quantitation of the 2.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 2.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may precede using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance

- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use

his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning and Silanizing Procedure

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, “qualified” data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions

may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.
If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The five surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10, Pyrene-d10 and 1,4-Dioxane-d8) must meet the current statistically derived or nominal acceptance limits. If statistical limits have not been established, then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Non-Conformance Report (NCR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

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8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

For projects or clients requiring DoD QSM compliance, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, “Generation and Implementation of Statistical QC Limits and/or Control Charts”, current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All the compounds of interest must fall within the established statistical limits or nominal limits with the following sporadic exceedance allowances, for DoD clients.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For non-DoD clients, corrective action is only taken if greater than 10% of the analytes of interest are outside of the laboratory established acceptance limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

All MS/MSD samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

8.6 Contingency for handling out-of-control or unacceptable data – Contact Department Manager, Project Manager or Quality Assurance Officer to determine the contingency plan for out-of-control or unacceptable data. A Non-conformance Report or Corrective Action Report may need to be initiated.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8270C and Method 8270D.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

Katahdin SOP QA-808, “Generation and Implementation of Statistical QC Limits and/or Control Charts,” current revision.

LIST OF TABLES AND FIGURES

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. % Error must be ≤ 30%. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte = 15%; Option 2: linear least squares regression for each analyte: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-16	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
1,4-Dioxane-d8 (surrogate)
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichlorbenzene
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
1,2,4-trichlorobenzene
1,2,4,5-tetrachlorobenzene
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene
2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane-d8	96	66
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
1,3-dichlorobenzene	146	148, 111
1,4-dichlorobenzene	146	148, 111
1,2-dichlorobenzene	146	148, 111
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
1,2,4-trichlorobenzene	180	182, 145
1,2,4,5-tetrachlorobenzene	216	214, 179
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162
3-Nitroaniline	138	65,92

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5975B - N

DATE OF DFTPP INJECTION: 080718

JOB	SAMPLE	DATAFILE	DF	VIAL #	METHOD	UL INJ	CHEMIST	COMMENTS
W6283644-1	50ng DFTPP	ND575	1	1	DFTPPSIM	2.0	JLL	ICV
-4	SSTD 2.0 N0807	N9904		2	NSPSIM15			✓
-2	0.2	05		3				✓
-3	0.5	06		4				✓
-5	7.0	07		5				✓
-6	10.	08		6				✓
-7	15.	09		7				✓
-8	IMB	10		8				
								VICO807.K

STANDARD	CODE
DFTPP	53129
CAL. STD.	53123 53135 53146 53143
ICV. STD.	53145
IS MIX	53141

REVIEWED AND APPROVED BY:
DATE:

SVOA-005 – Revision 2 – 02/13/2018

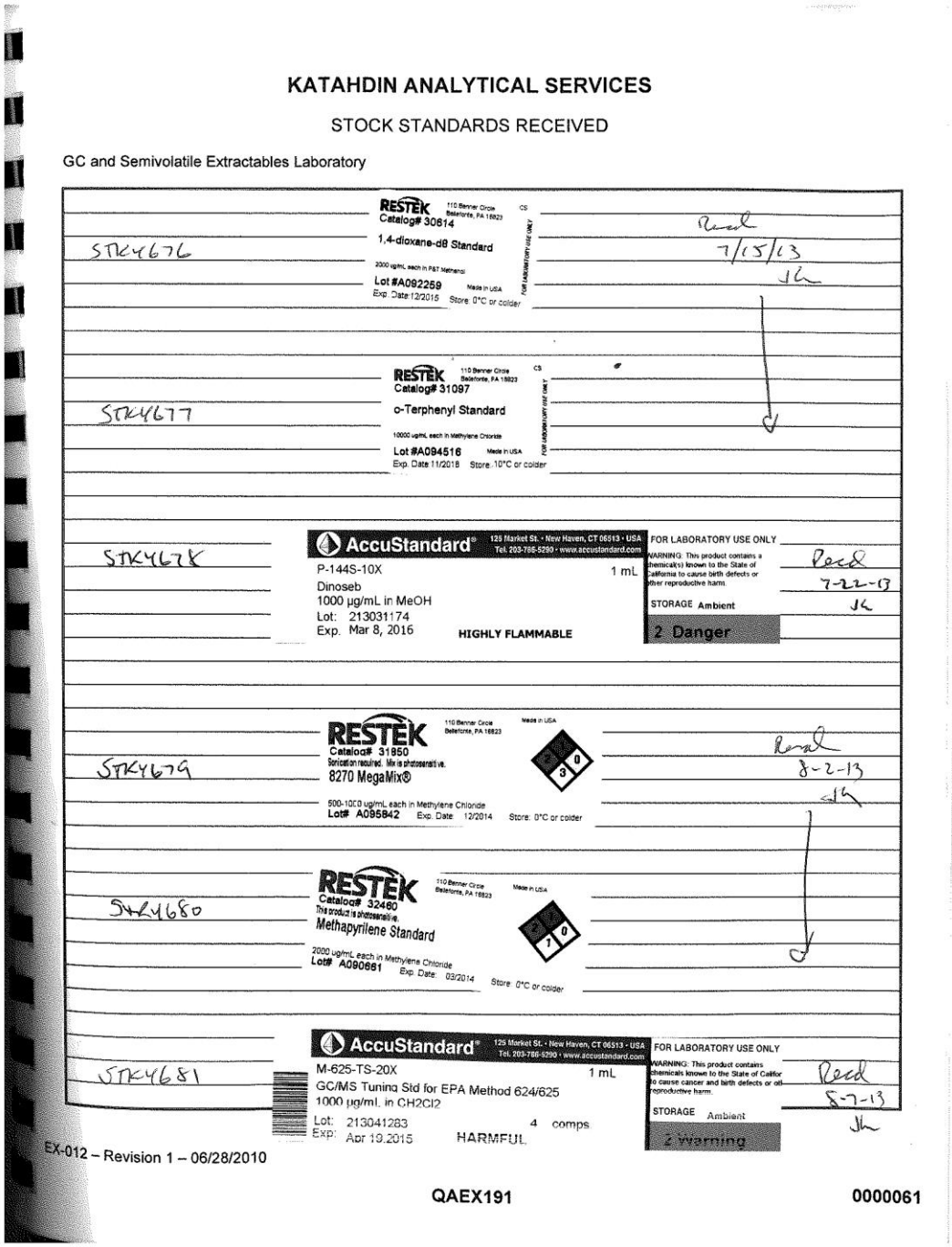
QAMS650

0000098

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GC and Semivolatile Extractables Laboratory

STK4676 — RESTEK Catalog# 30614, 1,4-dioxane-d8 Standard, Lot #A092259, Exp. Date 12/2015, Store: 0°C or colder. Rec'd 7/15/13 JK

STK4677 — RESTEK Catalog# 31097, o-Terphenyl Standard, Lot #A094516, Exp. Date 11/2018, Store: 10°C or colder

STK4678 — AccuStandard® P-144S-10X, Dinoseb, 1000 µg/mL in MeOH, 1 mL, Lot: 213031174, Exp. Mar 8, 2016, HIGHLY FLAMMABLE, FOR LABORATORY USE ONLY, STORAGE Ambient, 2 Danger. Rec'd 7-22-13 JK

STK4679 — RESTEK Catalog# 31850, Sonication required. Mix is photosensitive. 8270 MegaMix®, 500-1000 µg/mL each in Methylene Chloride, Lot# A095842, Exp. Date 12/2014, Store: 0°C or colder. Rec'd 8-2-13 JK

STK4680 — RESTEK Catalog# 32460, This product is photosensitive. Methapyrilene Standard, 2000 µg/mL each in Methylene Chloride, Lot# A090661, Exp. Date: 03/2014, Store: 0°C or colder

STK4681 — AccuStandard® M-625-TS-20X, GC/MS Tuning Std for EPA Method 624/625, 1000 µg/mL in CH2Cl2, 1 mL, Lot: 213041283, Exp: Apr 19,2015, 4 comps, HARMFUL, FOR LABORATORY USE ONLY, STORAGE Ambient, 2 Warning. Rec'd 5-7-13 JK

EX-012 – Revision 1 – 06/28/2010

QAEX191

0000061

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

SVOA-004 – Revision 1 – 11/10/10

GC/MS SVOA STANDARD PREP LOGBOOK

Standard No.	Standard Name	Prep Date	Exp. Date	Initials	Stock #	Stock Name	Amt Added	Stock Exp Date	Total Vol	Final Conc
S2401	SIM Stock B	4-11-14	6-14-14	JLM	S2367	SIM Pre-Stock B	180	6-14-14	1.8 ml	20 µg/ml
					DK009	MeCl2	1620			
S2402	SIM IND	4-15-14	10-15-14	JLM	STD5510	Acid S	~~450~~ 400	4-7-15	1.0ml	100 µg/ml
	Pre Stock 2				STD5298	B/N	50			
					STD5516	1-Meth Napht				
					STD5348	Benzene, 3,5-Dich				
					STD 5316	Component B3				
					STD 5305 5305	1,4-Dioxane				
					STD 5345	OLM	20			
					DK009	MeCl2	680			
					STD5317	1,2,4-5-TCB2	100 JLM			
S2403	SIM IND	4-15-14	10-15-14	JLM	S2402	SIM Pre Stock 2	100	10-15-14	1.0ml	10 µg/ml
	Pre Stock 1				DK009	MeCl2	900			
S2404	SIM IND	4-15-14	10-15-14	JLM	S2403	SIM Pre Stock 1	200	10-15-14	1.0 ml	2 µg/ml
					DK009	MeCl2	800			

Reviewed by/Date:

CAMS521

0000027

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

ADDENDUM 1

LOW LEVEL 1,4-DIOXANE ANALYSIS

The following are differences from the standard 8270 C or D SIM analysis:

GC Operating Conditions – The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP (Sect. 7.4) with the following exceptions:

Column Temperature Program	35°C hold 3 minutes 20°/min. to 300°C
Final Column Temperature hold	300°C
Run Time	16.25 minutes
Scan Start Time	2.3 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

Stock Standards – 1,4-Dioxane and 1,4-Dioxane each at 20 ug/mL

Calibration Standards – Use the above stock standards to prepare calibration standards at concentrations 0.25 ug/mL, 0.50 ug/mL, 1.0 ug/mL, 2.0 ug/mL, 4.0 ug/mL and 6.0 ug/mL. The 1.0 ug/mL is also the continuing calibration verification standard.

Sample analysis – Add 1 uL of internal standard (Section 5.3.2.4) aliquot of sample.

The ions for 1,4-Dioxane are 58 and 88.
The ions for 1,4-Dioxane-d8 are 64 and 96.

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DN	1.23.01	12301
02 5035	Reorganiced Sections 4, 5, 6, 7 and 8.	HRC	07.02.09	07.02.09
03 5035	Editted Section 6.4.3 to include the addition of 5mL of H2O to Sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06
05	Added 3585 Reference. Sections 6.1.2.3, 6.4.3 and 7.2.2: changed 20mL to 5mL.	LAD	09108	09/08

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Small changes to sections 3.2, 4.2.2.1, and 7.1.1 to address the differences between Purge and Trap Autosamplers. Added references.	LAD	03/12	03/12
07	Sec 6.1.1.2 - Changed 20mL to 5mL. Secs 6.1, 6.2, 6.4: edited and added steps for clarity. Changed required balance accuracy from 0.1 g to 0.01g throughout. Minor formatting and grammatical changes. Updated references	LAD	03/18	03/18
08	Changed 200 ug/kg to 150 ug/kg. Changed some DI H2O to read DI H2O. Sect. 4.2.1 - updated purge instrument requirements. Other minor edits.	LAD	01/20	01/20

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-08**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-08**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 150 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent by method 3585. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 5030, 5035 and 3585. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 150 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 150 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 150 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 40 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared in n-hexandecane according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free laboratory

reagent grade water or water miscible solvent and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Purge and Trap autodampler systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the

sample. The device should also be capable of introducing at least 5 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240°C - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 29 - 30 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.3 Micro syringes - 10-, 100-µL.

4.3.4 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.1 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS AND STANDARDS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low concentration soil samples

Sodium bisulfate preservation is used in the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

Water and subsequent freezing preparation of vials is used in the collection of low concentration soil samples known to contain carbonate minerals which may effervesce upon contact with an acidic preservation solution and which are to be analyzed by the closed-system purge-and-trap equipment described in Method 5035. This type of preservation is typically done in the lab after Encore samplers are received from the field. This must be done within 48 hours of sampling.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 The preservative is added to each vial prior to shipping the vial to the field. Add 5 mL of 20% sodium bisulfate solution or 5 mL of water to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is

negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.1 g and record it in the logbook.

6.1.2 High concentration soil samples in methanol:

6.1.2.1 When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40-mL glass vials with septum seals (see Sec. 4.4).

6.1.2.2 The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.2.3 Add 5 mL of methanol to each vial.

6.1.2.4 Seal the vial with the screw-cap and septum seal.

6.1.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.2.6 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.2.2, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.1.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™ , and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 5 mL of water and storage at -10° (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples.

7.0 PROCEDURES

This section describes procedures for the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

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For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.1 Low concentration soil method (Approximate concentration range of 5 to 150 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Purge and Trap Autosampler Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

7.1.1.1 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.2 Carry out the purge-and-trap procedure as outlined in Secs. 7.1.2. to 7.1.4.

7.1.2 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain sealed in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial

in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water, the internal standards, and the surrogate compounds. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

7.1.3.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 1 to 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration method for soil samples with concentrations generally greater than 150 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 5.0 g (wet weight) of sample to a tared 40-mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.1 g. Add 5 mL of methanol to the vial containing the sample and shake for two minutes.

NOTE: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.3 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.4 For each new lot of methanol, add an appropriate aliquot of the methanol to 40 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared

using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane and shake for two minutes.

7.3.2 For oily samples that are soluble in methanol if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), V (2015) and VI, Method 5035

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), V (2015) and VI, Method 5035A

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 3585, SW-846, USEPA, Revision IIIB, Nov. 2004.
Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM, Current Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

LIST OF TABLES AND FIGURES

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-08	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: James Sampson

Review Date: 5-7-21

SOP Number: CA-214

SOP Title: Closed system purge and trap method 5035

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:** 5-12-21

QAO Signature: Susan Dimond **Date:** 051221

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 9-30-2022

SOP Number: CA-214-08

SOP Title: Closed System Purge + Trap + Extraction for Volatile organics in Soil + Waste Samples by SW 846 Method 5035

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:** 9-30-2022

QAO Signature: Leslie Dimond **Date:** *Oct 19, 2022*

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

Prepared By: GC/MS Group Date: 07/01

Approved By:

Group Supervisor: [signature] Date: 080307

Operations Manager: John C. Burton Date: 08/03/01

QA Officer: Deborah J. Nadeau Date: 8/6/01

General Manager: Desmond F. Hurley Date: 8/6/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8260B	Minor change to criteria in section 7.5.4. Updated calibration standard mixes.	EN	5.23.02	5.23.02
02 8260B	Reorganization of sections 4,5,6 and 7 and Tables and Figures. Added definitions and information for the new data processing system.	MRC	07.06.04	07.06.04
03 8260B	Corrected %RSD for Initial Cal. in Sect. 7.4.3 & Table 1 added wording to Sec.s 6 and 8 minor changes throughout	LAD	020405	020405
04 8260B	Change Title to include all VOC's. Added instrumentation to sect. 4.4, 7.4.2 & 7.6.3. Update std. conc. sect's 5.3.2.1, 5.3.2.2, 5.3.2.3, 7.4.4, 7.5.1 & 7.5.9. Added "T" instr. and removed "Q" instr. Updated cal. information. added compounds to Table 3. Updated IS and SS mixes.	LAD	04/06	04/06
05	Sect. 4.7 - changed syringe sizes Sect. 5.2 - changed milli Q to Siemens Sect. 7.3 - changed reset time sect. 7.4.1 - Changed desorb time Added ICV criteria, MI references and RT window criteria.	LAD	06/07	06/07

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 4.7 - changed syringe size Sect. 6.2 - Changed Milli-Q to Siemens Sect. 7.3 - changed Runtime Sect. 7.4.1 - Changed desorb time Added ICV criteria, MI references and RT window criteria	LAD	06/07	06/07
07	Updated sections 7.4.4, 7.4.6, 7.5.2, 8.1, 8.2, 8.3, 10.0 and Table 1 with DoD QSM Version 4.1 criteria.	LAD	08/09	08/09
08	Removed the "Z" instrument and added the "C" and "D" instruments. Added Table 2 - DoD QSM ver. 4.1 QC Requirements.	LAD	04/10	04/10
09	Removed Tekmar 2000 and 2016 throughout. Added heated purge and 1,4-Dioxane information. Update standard prep information, columns, instrument configuration, purge and desorb conditions to reflect current practices.	LAD	03/12	03/12
10	Throughout - minor edits for reporting through KIMS and the QSM version number.	LAD	04/13	04/13
11			LAD 09/13	
11	Sect. 9, Tables 4 & 5 - Updated to include Carbon Disulfide, m, p, o-Xylenes, Ethyl-benzene and methylcyclohexane. Updated Figures 1→3	LAD	09/13	09/13
12	Sect. 5 - added Standards to title. Sect. 7 - minor edits to reflect current practices. Added Table 3 - DoD QSM 5.0 QC Requirements, renumbered rest. Also fixed Table references	LAD	05/16	05/16

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
13	Added Methylene chloride and Toluene, Added new column information	LAD	09/16	09/16
14	Sect. 10 - Updated method references. Sect. 4 - added GC + MS models. Sect 5 + 7 - added soil parameters for Initial Calibration, IS, SS + LCS standard prep.	LAD	09/17	09/17
15 LAD 3/5/19	Removed all Tekmar references. Changed independant standard expiration to 14 days. Added 2 ICAL levels. Updated references. Removed Table 2, DoD QSM 4.2 QC requirements. Updated logbook example. Updated Table 4 and 5 w/ current analyte list	LAD	03/19	03/19
16	Removed all soil, 1,4-Dioxane, heated purge and 5890 instrument references. Added 1,2,3-Trichlorobenzene. Updated Column to RTX-VMS, 30 M, 0.25mm ID	LAD	06/20	06/20

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-220-16**, titled **ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-220-16**, titled **ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

Recipient: _____ Date: _____

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze aqueous samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision, modified for selected ion monitoring to achieve lower detection levels for Volatile Organic Compounds (VOCs).

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC Volatile Organic Compounds

VOA Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination. When analyzed directly after a calibration, the LCS doubles as the Independent Calibration Verification (ICV).

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample prior to sample

analysis. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260 modified for SIM. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatiles organics by Method 8260, modified for SIM, to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention and Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual.

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous samples with helium, an inert gas, for a set period to efficiently transfer purgeable organics to the gaseous phase. These volatile organics are then retained on a cooled trap (SP1000/tenax/silica gel medium, or equivalent) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890 and 7890

4.2 Mass Spectrometers (MS): HP5972, HP5973 and HP5975.

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber components are not to be used.

4.4 Column: RTX-VMS, 30 meter, 0.25mm ID

4.5 Purge and Traps: Archon 5100 and Centurion auto samplers, and Encon concentrators.

4.6 Purge tube: 25 ml fritted purge vessel and 5ml fritted purge vessel for heated purge.

4.7 Hamilton Gastight and or SGE Gastight syringes: 5.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 Purge and trap grade methanol

5.2 Organic-free laboratory reagent grade water: Siemens Water Technologies. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

Ampulated standards must not be used past the manufacturer’s expiration date.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the components listed below. The final concentration of each component is 5.0 ug/mL (25 ug/mL for 4-methyl2-pentanone and 2-Hexanone). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

1,1,1,2-Tetrachloroethane	1,3-Dichlorobenzene	Hexachlorobutadiene
1,1,1-Trichloroethane	1,3-Dichloropropane	Isopropylbenzene
1,1,2,2-Tetrachloroethane	1,4-Dichlorobenzene	m,p-Xylene
1,1,2-Trichloroethane	2-Hexanone	Methylcyclohexane
1,1-Dichloroethane	4-Methyl-2-pentanone	Methylene Chloride
1,1-Dichloroethene	Benzene	o-Xylene
1,2,3-Trichlorobenzene	Bromodichloromethane	Tetrachloroethene
1,2,3-Trichloropropane	Carbon Disulfide	Toluene

1,2,4-Trimethylbenzene	Carbon Tetrachloride	trans-1,2-Dichloroethene
1,2,4-Trrichlorobenzene	Chloroform	trans-1,3-Dichloropropene
1,2-Dibromo-3-chloropropane	Chloromethane	Trichloroethene
1,2-Dibromomethane	Cis-1,2-Dichloroethene	Trichlorofluoromethane
1,2-Dichlorobenzene	cis-1,3-Dichloropropene	Vinyl Chloride
1,2-Dichloroethane	Dibromochloromethane	Xylenes
1,2-Dichloropropane	Ethylbenzene	

5.3.2.2 Laboratory Control Spike and MS/MSD Mixture - Prepare a standard independent from the calibration standards, as above containing the compounds listed below. The final concentration of each component is 5.0 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

1,1,1,2-Tetrachloroethane	1,3-Dichlorobenzene	Hexachlorobutadiene
1,1,1-Trichloroethane	1,3-Dichloropropane	Isopropylbenzene
1,1,2,2-Tetrachloroethane	1,4-Dichlorobenzene	m,p-Xylene
1,1,2-Trichloroethane	2-Hexanone	Methylcyclohexane
1,1-Dichloroethane	4-Methyl-2-pentanone	Methylene Chloride
1,1-Dichloroethene	Benzene	o-Xylene
1,2,3-Trichlorobenzene	Bromodichloromethane	Tetrachloroethene
1,2,3-Trichloropropane	Carbon Disulfide	Toluene
1,2,4-Trimethylbenzene	Carbon Tetrachloride	trans-1,2-Dichloroethene
1,2,4-Trrichlorobenzene	Chloroform	trans-1,3-Dichloropropene
1,2-Dibromo-3-chloropropane	Chloromethane	Trichloroethene
1,2-Dibromomethane	Cis-1,2-Dichloroethene	Trichlorofluoromethane
1,2-Dichlorobenzene	cis-1,3-Dichloropropene	Vinyl Chloride
1,2-Dichloroethane	Dibromochloromethane	Xylenes
1,2-Dichloropropane	Ethylbenzene	

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5.3.2.3 Internal Standard and Surrogate spiking solution for 8260 SIM – Prepare a combined standard as above containing the compounds listed below. The final concentration of each component is 25 ug/mL. The standard must be prepared every 14 days and stored on the Auto Sampler in a pressurized vial or in the VOA standards freezer between uses.

Internal Standard	**Surrogate**
Pentafluorobenzene	Dibromofluoromethane
1,4-Difluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene

5.3.2.4 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared

every 30 days and stored in the VOA standards freezer between uses.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are sampled into 40 mL glass VOA vials leaving no headspace. Samples are preserved with 2 drops 1:1 HCL and stored at 4°C (±2°C) until analysis.

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files:

BFB Tune: VOABFBAQ.M

All other samples and standards:YSIMAXX.M

where: Y = instrument ID (Each instrument is given a unique identifier)
XX = the calibration number in chronological order

Data files:

BFB: IB_ _ _.D
where: I is the instrument ID
_ _ _ is a number in chronological order from 000 to 999

All other data files: I_ _ _ _.D
Where: I is the instrument ID
_ _ _ _ is a number in chronological order from 0000 to 9999

This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

Column:	RTX-VMS, 30meter, 0.25mm ID. or equivalent.	
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:	150°	
Run time:	6-8 minutes	
Scan start time:	3 minutes	
Scan parameters:	not to exceed 2 sec per scan	
Mass range:	35-300	
Number of A/D samples:	8	
GC peak threshold:	1000 counts	
Threshold:	100 counts	

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 8260 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-

injected and reevaluated. If the instrument still does not meet criteria, notify your department manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Archon 5100, Setup/Operation: Please refer to the Archon Manual for more detailed operations for these instruments.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon, the settings for these features will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:	Purge Gas: Helium
	Purge Time: 11.0 ±0.1 minute
	Purge Flow Rate: appox. 40 mL/min
	Purge Temperature: Ambient (water)
Desorb Conditions:	DesorbTemp: 250ºC
	Desorb Flow rate: 15mL/min
	Desorb Time: 1.0-2.0± 0.1 min
	Bake Time: 10 min
	Bake Temperature: 260º C

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method 8260 SIM

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

The GC/MS must be initially calibrated at six different levels (target compounds at concentrations listed below) to determine the linearity of response. See Section 5.3.2 for preparation of the calibration standards. Tables 4 and 5 contain a list of target compounds, internal standards, and surrogates with their defined primary quantitation ions.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

SIM 8260 AQ	
8260 Sim	**Cal mix @ 5.0/25 ug/mL**
VSTD0.05	1.0 uL
VSTD0.075	1.5 uL
VSTD0.10	2.0 uL
VSTD0.30	6.0 uL
VSTD0.50	10 uL
VSTD0.75	15 uL
VSTD1.00	20 uL
VSTD2.00	40 uL

The internal standard and surrogates are spiked by the autosampler.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 4 and 5.

7.4.4. Initial Calibration Criteria

Refer to Tables 1 & 2, QC Requirements, for specific criteria that must be met for Method 8260. The percent (%) RSD for VOCs must be less than or equal to 15% (30% for heated purge).

For projects or clients requiring DoD QSM, current version, an independent calibration verification (ICV) sample must be run. The ICV must contain compounds from a different source than the ICAL. All project analytes must

fall between 80-120% of the true value. No samples may be run until the ICV criteria is met.

7.4.5. Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing the target compounds, internal standards and surrogates at the concentrations below must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB.

SIM 8260	
Standard	**Conc.**
VOC Mix	0.5ug/mL
SS Mix	1.0 ug/mL
IS Mix	1.0 ug/mL

The relative response factor from the continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extended ion current profile) area for the internal standard in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for the internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for VOCs is less than or equal to 15% (30% for heated purge), the continuing calibration is considered valid.

Continuing calibration check criteria must be met before sample analysis can proceed. The CV level standard analyzed as part of the initial calibration curve can be used as the continuing calibration standard, assuming all criteria are met, and time is left in the twelve-hour window to analyze samples.

7.4.6. Retention Time Windows

Retention time windows for the internal standards are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows for the internal standards of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The internal standard of the samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

For projects or clients requiring DoD QSM, current version, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

7.5 Quality Control Sample Analysis

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1. Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the AQ LCS, 10 uL of the LCS standard mix at 5.0 ug/mL is spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 0.5 ug/L.

The autosampler adds the internal and surrogate standard to a 25mL aliquot of this preparation for analysis. The concentration of the IS/SS mix is dependant of which autosampler is being used.

If the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2. Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the 1/10th the measured amount in any sample or 1/10th the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3. Surrogate Recovery Limits

For blanks, laboratory control samples, and client samples, the nominal limits must be met for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4. Internal Standard Area Recoveries / Retention Times.

The internal standard response and retention time in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extended ion current profile) area for the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for the internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must by inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

7.5.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 25 mL aliquots (aqueous), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration equal to the mid point calibration level. Acceptance criteria for MS/MSD pairs are outlined in Section 8.0.

If sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. They may be able to provide more information about

the sample. Sample history is used to determine what order in which to run the samples and at what dilution.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

7.6.2 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a twelve-hour window. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

For All Units:

Record the sample pH in the injection logbook.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria tables found within this SOP (Table 1 & 2). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or internal standard area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalysis.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. A “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the department manager or his/her designee, who will review each manual integration.

For specific Manual Integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form I as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the GC/MS Department Manager, or his designee.

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics Department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Tables 1 & 2 and to details in this section for a summary of QC requirements, acceptance criteria and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client, and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases, “qualified” data with narration may be advisable after consultation with the client.

In some cases, the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases, the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 LCS Criteria

Nominal limits of 70-130% (60-140% for heated purge) are used. Where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than $\pm$ 3 standard deviations from the mean recovery.

8.2 MS/MSD Criteria

Nominal limits of 70-130% (60-140% for heated purge) are used. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than $\pm$ 3 standard deviations from the mean recovery. A MS/MSD must be analyzed with each analytical/preparatory batch per matrix. RPD must be $\leq$ 30% between the MS and MSD.

8.3 Surrogate Recovery Criteria

Surrogate Limits: (Nominal Limits) 70-130% (60-140% for heated purge).

For projects or clients requiring DoD QSM, current version, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used.

8.4 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases, data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if

the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client, and project specific Data Quality Objectives and on review of chromatograms. The department manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases, "qualified" data with narration may be advisable after consultation with the client.

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.

The Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.
Refer to the current revision of Method 8260 for other method performance parameters and requirements.

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8260B.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8260C.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8260D.

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.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration	Initial calibration prior to sample analysis	RSD <15%	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances	If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Calibration verification	Once per each 12 hours, prior to sample analysis	RF within 15% of average initial multi-point RF.	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for IS.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples For projects requiring DoD QSM, one per preparatory batch.	No analytes of interest detected > PQL See section 7.5.2 of this SOP for additional DoD acceptance requirements.	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Nominal 70-130% recovery (60-140% for heated purge) For projects requiring DoD QSM, DoD limits shall be used, if available. Otherwise lab limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out re-extract and analyze sample (4) If reanalysis is out, flag data

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One LCS per batch of 20 or fewer samples	70-130% Recovery (60-140% for heated purge)	(1) Evaluate the samples and associated QC: If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
MS/MSD	One MS/MSD per every 20 samples	Nominal limits of 70-130% (60-140% for heated purge) are used as default limits. See also section 8.2 of this SOP.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		
Demonstration of Proficiency	Once per analyst initially; 4 reps of LCS and annually thereafter	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation = 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte = 15%; Option 2: linear least squares regression for each analyte: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-220-15	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(4) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC – Spikes	None	
QC – LCS	None	
QC – Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC – MDL	None	

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 4

CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,1-Trichloroethane	97	99, 61
1,1,2,2-Tetrachloroethane	83	131, 85
1,1,2-Trichloroethane	83	97, 85
1,1-Dichloroethane	75	53, 77
1,1-Dichloroethene	96	61, 63
1,2,3-Trichlorobenzene	180	182, 145
1,2,3-Trichloropropane	75	77, 110
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-chlororpropane	75	155, 157
1,2-Dibromomethane	107	109, 188
1,2-Dichlorobenzene	146	111, 148
1,2-Dichloroethane	62	98
1,2-Dichloropropane	63	112
1,3-Dichlorobenzene	146	111, 148
1,3-Dichloropropane	76	78
1,4-Dichlorobenzene	146	111, 148
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58
Acrylonitrile	53	52, 51
Benzene	78	77, 51
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
cis-1,2-Dichloroethene	96	61, 98
cis-1,3-Dichloropropene	75	77, 39
Dibromochloromethane	129	127
Dibromomethane	93	95, 174
Ethylbenzene	91	106
Hexachlorobutadiene	225	223, 227
Isopropylbenzene	105	120
m,p-Xylene	91	106
Methyl tert-butyl ether	73	57, 41
Methylcyclohexane	83	55, 98
Methylene chloride	84	86, 49
o-Xylene	91	106
Tetrachloroethene	164	129, 131
Toluene	92	91
Trans-1,2-Dichloroethene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Trichloroethene	95	97
Trichlorofluoromethane	101	103
Vinyl Chloride	62	64
Xylenes	91	106
Naphthalene	128	-

TABLE 5

INTERNAL STANDARD

INTERNAL STANDARD	TARGET ANALYTE
Pentafluorobenzene	1,1,1-Trichloroethane
	1,1-Dichloroethane
	1,1-Dichloroethene
	Acrylonitrile
	Bromomethane
	Carbon disulfide
	Chloroethane
	Chloroform
	Chloromethane
	Cis-1,2-Dichloroethene
	Methyl tert-butyl ether
	Methylcyclohexane
	Methylene chloride
	trans-1,2-Dicholoethene
	Trichlorofluoromethane
	Vinyl Chloride
INTERNAL STANDARD	**TARGET ANALYTE**
1,4-Difluorobenzene	1,1,2-Trichloroethene
	1,2-Dibromomethane
	1,2-Dichloroethane
	1,2-Dichloropropane
	4-Methyl-2-pentanone
	Benzene
	Bromodichloromethane
	Carbon Tetrachloride
	cis-1,3-Dichloropropene
	Dibromomethane
	Toluene
	Trans-1,3-Dichloropropene
	Trichloroethene
INTERNAL STANDARD	**TARGET ANALYTE**
Chlorobenzene-d5	1,1,1,2-Tetrachloroethane
	1,3-Dichloropropane
	2-Hexanone
	Chlorobenzene
	Dibromochloromethane
	Ethylbenzene
	m+p-Xylene
	o-Xylene
	Tetrachloroethene
INTERNAL STANDARD	**TARGET ANALYTE**
1,4-Dichlorobenzene-d4	1,1,2,2-Tetrachloroethane
	1,2,3-Trichloropropane
	Isopropylbenzene
	1,2,4-Trimethylbenzene
	Bromoform
	1,3-Dichlorobenzene
	1,4-Dichlorobenzene
	1,2,3-Trichlorobenzene
	1,2-Dichlorobenzene
	1,2-Dibromo-3-chlororpropane
	Hexachlorobutadiene
	1,2,4-Trichlorobenzene
	Naphthalene

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

W6

KATAHDIN ANALYTICAL SERVICES

GCMS-D INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 3-11-19 9:35

Method - Check One:	8260		8260 - 10 mL	8260 SIM ✓			624		524
STD IDs: *	BFB STD: V1412		CAL STD: V1401	GAS STD: —			XTRAs STD: —		LCS/MS STD: V1401
SURR STD: V1390			IS STD: V1390						

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	Prep Method 5030	Prep Method 5035	Prep Method 1311	Criteria KAS	Criteria DoD	Criteria QAPP	pH Paper Lot #: Y/N	pH Paper Lot #: ANALYST	pH Paper Lot #: pH	pH Paper Lot #: TRC	KI Paper Lot # COMMENTS
WG248398 -3	DB436	—	—	VOABFBAQ	✓						Y	JP/HC	N/A	N/A	
Vstd005 -4	D5856	1	1	DB2SIMDMH1							Y				
LCS -1	57		2								Y				
VBLKA	58		3								N				b-2ltc
VBLK13 -2	59		4								Y		↓		
SM1849 -2DA C	60		5						5.1		Y		<2		
-1DA C	61		6								Y		<2		
SM1905 -1BA H	62		7								Y		<2		
SM1849 -8MSBA H	63		8								Y		<2		
-8MSDBA G	64		9						↓		Y		<2		
CCV	65		10								Y		N/A		1 in SIM ✓
CCV	66		11								N				Not used
CCV	67		12								N				↓
Rinse	↓ 68	↓	13	✓	↓						—	↓	↓	↓	

3-12-19

* Refer to page 1 for standard preparation instructions.

VOA-012 - Revision 3 - 11/12/2018

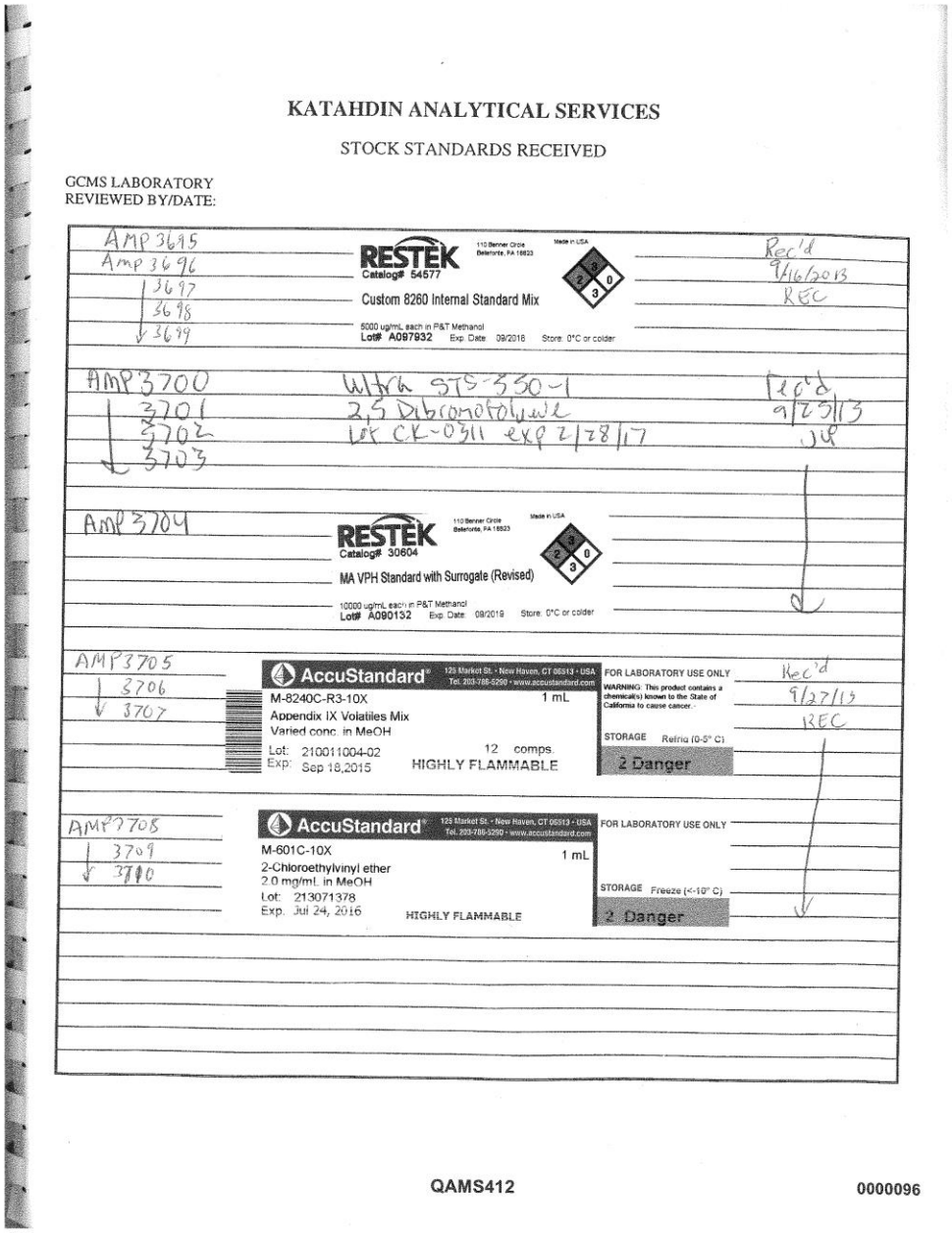
QAMS669

0000090

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP 3695
Amp 3696
3697
3698
3699

RESTEK 110 Benner Circle Bellefonte, PA 16823 Made in USA
Catalog# 54577
Custom 8260 Internal Standard Mix
5000 ug/mL each in P&T Methanol
Lot# A097932 Exp. Date: 09/2016 Store: 0°C or colder

Rec'd 9/16/2013 REC

AMP 3700
3701
3702
3703

Wtrk STS-330-1
2,5 Dibromotoluene
Lot CK-0311 exp 2/28/17

Rec'd 9/23/13 JG

AMP 3704

RESTEK 110 Benner Circle Bellefonte, PA 16823 Made in USA
Catalog# 30604
MA VPH Standard with Surrogate (Revised)
10000 ug/mL each in P&T Methanol
Lot# A090152 Exp. Date: 09/2016 Store: 0°C or colder

AMP 3705
3706
3707

AccuStandard® 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-8240C-R3-10X 1 mL
Appendix IX Volatiles Mix
Varied conc. in MeOH
Lot: 210011004-02 12 comps.
Exp: Sep 18,2015 HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.
STORAGE Refrig (0-5° C)
2 Danger

Rec'd 9/27/13 REC

AMP 3708
3709
3710

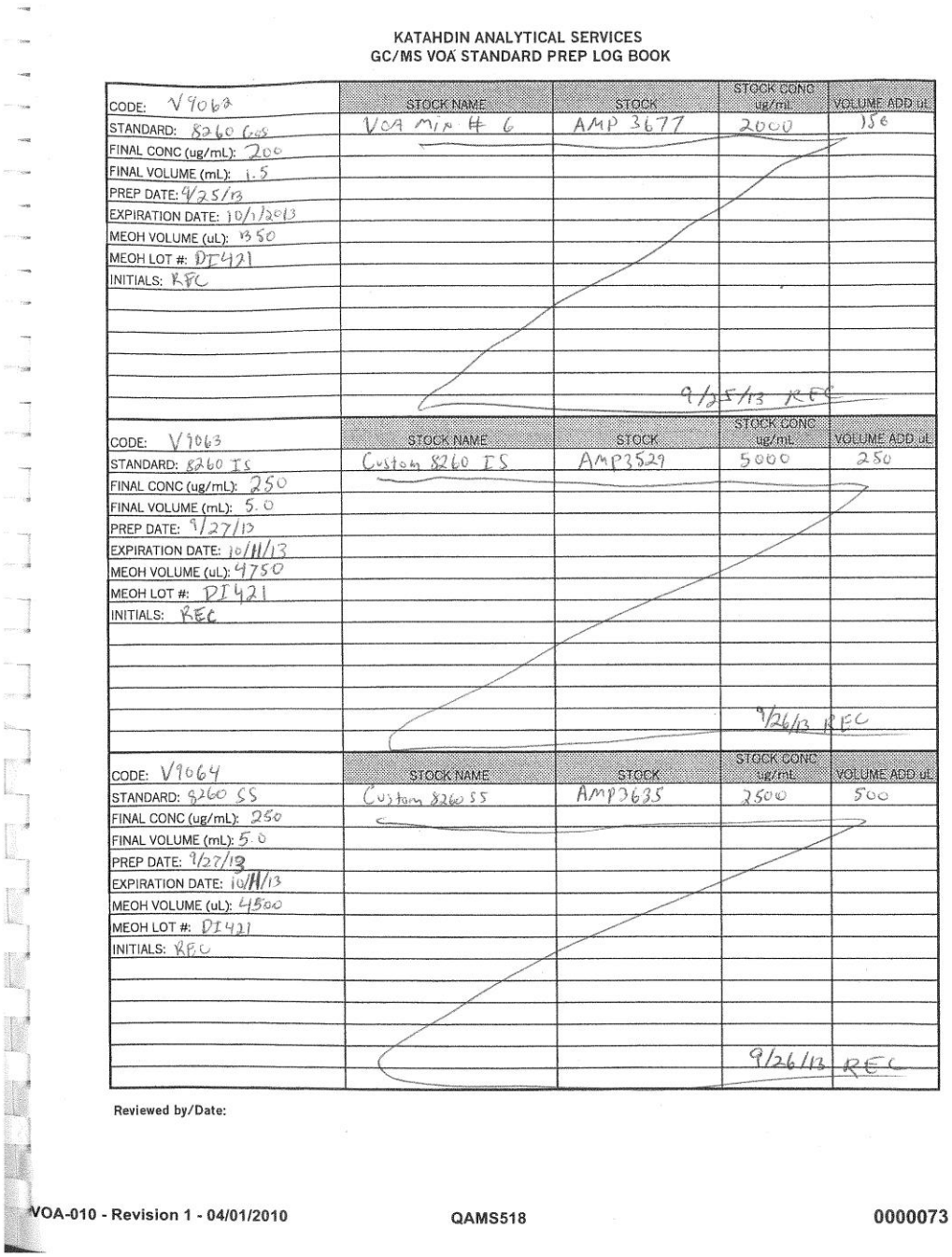
AccuStandard® 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com
M-601C-10X 1 mL
2-Chloroethylvinyl ether
2.0 mg/mL in MeOH
Lot: 213071378
Exp. Jul 24, 2016 HIGHLY FLAMMABLE
FOR LABORATORY USE ONLY
STORAGE Freeze (<-10° C)
2 Danger

QAMS412 0000096

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V9063	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 GAS	VOA Mix # 6	AMP 3677	2000	150
FINAL CONC (ug/mL): 200				
FINAL VOLUME (mL): 1.5				
PREP DATE: 9/25/13				
EXPIRATION DATE: 10/1/2013				
MEOH VOLUME (uL): 1350				
MEOH LOT #: DI421				
INITIALS: RFC				
		9/25/13 REC		

CODE: V9063	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 IS	Custom 8260 IS	AMP3529	5000	250
FINAL CONC (ug/mL): 250				
FINAL VOLUME (mL): 5.0				
PREP DATE: 9/27/13				
EXPIRATION DATE: 10/11/13				
MEOH VOLUME (uL): 4750				
MEOH LOT #: DI421				
INITIALS: REC				
		9/26/13 REC		

CODE: V9064	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 SS	Custom 8260 SS	AMP3635	2500	500
FINAL CONC (ug/mL): 250				
FINAL VOLUME (mL): 5.0				
PREP DATE: 9/27/13				
EXPIRATION DATE: 10/11/13				
MEOH VOLUME (uL): 4500				
MEOH LOT #: DI421				
INITIALS: REC				
		9/26/13 REC		

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS518 0000073

Appendix C: LOQ Verification Ranges

Katahdin Analytical Services
LOQ Verification

Method:	SW8260		Method:	SW8260	
Target List	AQ LOQ Lower Limits	AQ LOQ Upper Limits	Target List	SL LOQ Lower Limits	SL LOQ Upper Limits
Dichlorodifluoromethane	10	200	Dichlorodifluoromethane	10	200
Chloromethane	37	145	Chloromethane	37	145
Vinyl Chloride	42	153	Vinyl Chloride	42	153
Bromomethane	31	161	Bromomethane	31	161
Chloroethane	18	192	Chloroethane	18	192
1,1-Dichloroethene	75	140	1,1-Dichloroethene	75	140
Carbon Disulfide	52	149	Carbon Disulfide	52	149
Methylene Chloride	53	200	Methylene Chloride	54	200
Acetone	26	209	Acetone	26	209
trans-1,2-Dichloroethene	62	141	trans-1,2-Dichloroethene	62	141
Tertiary-butyl alcohol	10	198	Tertiary-butyl alcohol	10	198
1,1-Dichloroethane	58	148	1,1-Dichloroethane	58	148
cis-1,2-Dichloroethene	72	135	cis-1,2-Dichloroethene	72	135
Chloroform	62	144	Chloroform	62	144
1,1,1-Trichloroethane	60	146	1,1,1-Trichloroethane	60	146
2-Butanone (MEK)	51	152	2-Butanone (MEK)	51	152
Benzene	76	126	Benzene	76	126
Trichloroethene	65	135	Trichloroethene	65	135
Bromodichloromethane	72	135	Bromodichloromethane	72	135
Toluene	73	129	Toluene	73	129
4-methyl-2-pentanone(MiBK)	70	135	4-methyl-2-pentanone(MiBK)	70	135
Dibromochloromethane	74	130	Dibromochloromethane	74	130
Ethylbenzene	80	121	Ethylbenzene	80	121
m+p-Xylene	66	135	m+p-Xylene	79	126
o-Xylene	63	140	o-Xylene	62	138
Isopropylbenzene	52	151	Isopropylbenzene	46	156
n-Propylbenzene	71	134	n-Propylbenzene	71	134
1,3,5-Trimethylbenzene	66	137	1,3,5-Trimethylbenzene	66	137
tert-Butylbenzene	63	138	tert-Butylbenzene	71	133
1,2,4-Trimethylbenzene	60	140	1,2,4-Trimethylbenzene	71	130
Sec-Butylbenzene	68	135	Sec-Butylbenzene	68	135
p-Isopropyltoluene	60	144	p-Isopropyltoluene	55	145
1,3-Dichlorobenzene	78	118	1,3-Dichlorobenzene	78	118
n-Butylbenzene	63	136	n-Butylbenzene	63	136
1,2-Dichlorobenzene	78	121	1,2-Dichlorobenzene	78	121
1,2,4-Trichlorobenzene	60	142	1,2,4-Trichlorobenzene	60	142
1,2,3-Trichlorobenzene	53	139	1,2,3-Trichlorobenzene	53	139

Katahdin Analytical Services
LOQ Verification

Method	SW8260-SIM	
Target List	AQ LOQ Lower Limits	AQ LOQ Upper Limits
Chloromethane	50	150
Vinyl Chloride	50	150
Chloroethane	50	150
Trichlorofluoromethane	50	150
1,1-Dichloroethene	50	150
Methylene chloride	50	150
trans-1,2-Dichloroethene	50	150
1,1-Dichloroethane	50	150
Acrylonitrile	50	150
cis-1,2-Dichloroethene	50	150
Chloroform	50	150
Carbon Tetrachloride	50	150
1,1,1-Trichloroethane	50	150
Benzene	50	150
1,2-Dichloroethane	50	150
Trichloroethene	50	150
Dibromomethane	50	150
1,2-Dichloropropane	50	150
Bromodichloromethane	50	150
cis-1,3-Dichloropropene	50	150
Toluene	50	150
4-methyl-2-pentanone(MiBK	50	150
Tetrachloroethene	50	150
trans-1,3-Dichloropropene	50	150
1,1,2-Trichloroethane	50	150
Dibromochloromethane	50	150
1,2-Dibromoethane (EDB)	50	150
2-Hexanone	50	150
Ethylbenzene	50	150
Chlorobenzene	50	150
1,1,1,2-Tetrachloroethane	50	150
m+p-Xylene	50	150
o-Xylene	50	150
Isopropylbenzene	50	150
1,2,3-Trichloropropane	50	150
1,1,2,2-Tetrachloroethane	50	150
1,2,4-Trimethylbenzene	50	150
1,3-Dichlorobenzene	50	150
1,4-Dichlorobenzene	50	150
1,2-Dichlorobenzene	50	150
1,2-Dibromo-3-Chloropropar	50	150
Hexachlorobutadiene	50	150
1,2,4-Trichlorobenzene	50	150
1,2,3-Trichlorobenzene	50	150
Naphthalene	50	150

Katahdin Analytical Services
LOQ Verification

Method	8270		Method	8270	
Target List	AQ LOQ Lower Limits	AQ LOQ Upper Limits	Target List	SL LOQ Lower Limits	SL LOQ Upper Limits
1,4-Dioxane	10	95	1,4-Dioxane	10	74
n-Nitrosodimethylamine	10	123	n-Nitrosodimethylamine	20	138
Pyridine	10	135	Pyridine	10	78
2-Picoline	*10*	*190*	2-Picoline	*10*	*190*
n-Nitrosomethylethylamine	*10*	*190*	n-Nitrosomethylethylamine	*10*	*190*
Methyl Methanesulfonate	*10*	*190*	Methyl Methanesulfonate	*10*	*190*
n-Nitrosodiethylamine	*10*	*190*	n-Nitrosodiethylamine	*10*	*190*
Ethyl Methanesulfonate	*10*	*190*	Ethyl Methanesulfonate	*10*	*190*
Benzaldehyde	10	200	Benzaldehyde	10	172
Aniline	10	75	Aniline	10	131
Phenol	10	108	Phenol	39	125
Bis (2-Chloroethyl) Ether	29	112	Bis (2-Chloroethyl) Ether	32	122
2-Chlorophenol	29	106	2-Chlorophenol	42	117
1,3-Dichlorobenzene	19	108	1,3-Dichlorobenzene	47	102
1,4-Dichlorobenzene	20	109	1,4-Dichlorobenzene	50	105
Benzyl Alcohol	10	152	Benzyl Alcohol	48	125
1,2-Dichlorobenzene	21	112	1,2-Dichlorobenzene	56	103
2,2'-Oxybis(1-chloropropane	23	119	2,2'-Oxybis(1-chloropropane	40	111
2-Methylphenol	20	104	2-Methylphenol	46	112
n-Nitrosopyrrolidine	*10*	*190*	n-Nitrosopyrrolidine	*10*	*190*
Acetophenone	32	120	Acetophenone	45	116
n-Nitrosomorpholine	*10*	*190*	n-Nitrosomorpholine	*10*	*190*
n-Nitroso-di-n-propylamine	22	116	n-Nitroso-di-n-propylamine	39	107
o-Toluidine	*10*	*190*	o-Toluidine	*10*	*190*
3&4-Methylphenol	10	104	3&4-Methylphenol	42	121
Hexachloroethane	11	110	Hexachloroethane	48	100
Nitrobenzene	33	110	Nitrobenzene	44	118
n-Nitrosopiperidine	*10*	*190*	n-Nitrosopiperidine	*10*	*190*
Isophorone	40	106	Isophorone	10	139
2-Nitrophenol	31	119	2-Nitrophenol	48	112
2,4-Dimethylphenol	39	98	2,4-Dimethylphenol	36	115
O,O,O-Triethylphosphorothio	*10*	*190*	O,O,O-Triethylphosphorothio	*10*	*190*
Bis(2-chloroethoxy)methane	20	117	Bis(2-chloroethoxy)methane	52	111
Benzoic acid	10	200	Benzoic acid	10	181
2,4-Dichlorophenol	27	126	2,4-Dichlorophenol	50	115
1,2,4-Trichlorobenzene	20	107	1,2,4-Trichlorobenzene	53	103
Naphthalene	35	102	Naphthalene	40	114
4-Chloroaniline	13	121	4-Chloroaniline	10	117
2,6-Dichlorophenol	24	130	2,6-Dichlorophenol	48	134
Hexachloropropene	*10*	*190*	Hexachloropropene	*10*	*190*
Hexachlorobutadiene	16	103	Hexachlorobutadiene	49	100
n-Nitroso-di-n-butylamine	*10*	*190*	n-Nitroso-di-n-butylamine	*10*	*190*
Caprolactam	10	125	Caprolactam	10	177
4-Chloro-3-methylyphenol	50	114	4-Chloro-3-methylyphenol	46	125
Isosafrole	*10*	*190*	Isosafrole	*10*	*190*
2-Methylnaphthalene	32	121	2-Methylnaphthalene	13	156
1-Methylnaphthalene	10	151	1-Methylnaphthalene	45	105
Hexachlorocyclopentadiene	8	85	Hexachlorocyclopentadiene	10	135
1,2,4,5-Tetrachlorobenzene	*10*	*190*	1,2,4,5-Tetrachlorobenzene	*10*	*190*
2,4,6-Trichlorophenol	39	127	2,4,6-Trichlorophenol	47	119
2,4,5-trichlorophenol	26	164	2,4,5-trichlorophenol	50	121
Safrole	*10*	*190*	Safrole	*10*	*190*
1,1'-Biphenyl	33	122	1,1'-Biphenyl	29	134

Katahdin Analytical Services
LOQ Verification

Method	8270		Method	8270	
Target List	AQ LOQ Lower Limits	AQ LOQ Upper Limits	Target List	SL LOQ Lower Limits	SL LOQ Upper Limits
2-Chloronaphthalene	24	89	2-Chloronaphthalene	56	114
1-Chloronaphthalene	*10*	*190*	1-Chloronaphthalene	*10*	*190*
2-Nitroaniline	38	125	2-Nitroaniline	38	141
1,4-Naphthoquinone	*10*	*190*	1,4-Naphthoquinone	*10*	*190*
Dimethyl phthalate	10	144	Dimethyl phthalate	47	140
1,3-Dinitrobenzene	*10*	*190*	1,3-Dinitrobenzene	*10*	*190*
2,6-Dinitrotoluene	49	125	2,6-Dinitrotoluene	49	124
Acenaphthylene	46	110	Acenaphthylene	42	125
3-Nitroaniline	30	114	3-Nitroaniline	10	200
Acenaphthene	45	112	Acenaphthene	51	119
2,4-Dinitrophenol	10	187	2,4-Dinitrophenol	10	101
Pentachlorobenzene	*10*	*190*	Pentachlorobenzene	*10*	*190*
4-Nitrophenol	10	163	4-Nitrophenol	14	170
Dibenzofuran	48	118	Dibenzofuran	45	123
2,4-Dinitrotoluene	47	142	2,4-Dinitrotoluene	48	127
1-Naphthylamine	*10*	*190*	1-Naphthylamine	*10*	*190*
2,3,4,6-Tetrachlorophenol	26	143	2,3,4,6-Tetrachlorophenol	46	105
2-Naphthylamine	*10*	*190*	2-Naphthylamine	*10*	*190*
Diethylphthalate	43	115	Diethylphthalate	42	131
Fluorene	49	122	Fluorene	40	125
O,O-Diethyl-o-2-pyrazinylpho	*10*	*190*	O,O-Diethyl-o-2-pyrazinylpho	*10*	*190*
4-Chlorophenyl-phenylether	54	111	4-Chlorophenyl-phenylether	52	125
5-Nitro-O-toluidine	*10*	*190*	5-Nitro-O-toluidine	*10*	*190*
4-Nitroaniline	34	124	4-Nitroaniline	10	176
4,6-Dinitro-2-methyphenol	26	154	4,6-Dinitro-2-methyphenol	17	134
n-Nitrosodiphenylamine	35	112	n-Nitrosodiphenylamine	10	189
1,2-Diphenylhydrazine	43	111	1,2-Diphenylhydrazine	31	142
Sulfotepp	*10*	*190*	Sulfotepp	*10*	*190*
1,3,5-Trinitrobenzene	*10*	*190*	1,3,5-Trinitrobenzene	*10*	*190*
Diallate	*10*	*190*	Diallate	*10*	*190*
Phorate	*10*	*190*	Phorate	*10*	*190*
Phenacetin	*10*	*190*	Phenacetin	*10*	*190*
4-Bromophenyl-phenylether	40	122	4-Bromophenyl-phenylether	49	136
Hexachlorobenzene	31	132	Hexachlorobenzene	49	128
Dimethoate	*10*	*190*	Dimethoate	*10*	*190*
Atrazine	60	176	Atrazine	29	161
4-Aminobiphenyl	*10*	*190*	4-Aminobiphenyl	*10*	*190*
Pentachlorophenol	11	165	Pentachlorophenol	34	148
Pentachloronitrobenzene	*10*	*190*	Pentachloronitrobenzene	*10*	*190*
Proamide	*10*	*190*	Proamide	*10*	*190*
Phenanthrene	58	119	Phenanthrene	42	136
Dinoseb	*10*	*190*	Dinoseb	*10*	*190*
Disulfoton	*10*	*190*	Disulfoton	*10*	*190*
Anthracene	53	126	Anthracene	36	134
Carbazole	35	148	Carbazole	10	200
Methyl parathion	*10*	*190*	Methyl parathion	*10*	*190*
Di-n-butylphthalate	52	129	Di-n-butylphthalate	31	154
4-Nitroquinoline-1-oxide	*10*	*190*	4-Nitroquinoline-1-oxide	*10*	*190*
Parathion	*10*	*190*	Parathion	*10*	*190*
Methapyrilene	*10*	*190*	Methapyrilene	*10*	*190*
Isodrin	*10*	*190*	Isodrin	*10*	*190*
Fluoranthene	50	137	Fluoranthene	55	131
Benzidine	10	200	Benzidine	10	200
Pyrene	32	161	Pyrene	29	156
Aramite	*10*	*190*	Aramite	*10*	*190*
p-Dimethylaminoazobenzene	*10*	*190*	p-Dimethylaminoazobenzene	*10*	*190*

Katahdin Analytical Services
LOQ Verification

Method	8270	
Target List	AQ LOQ Lower Limits	AQ LOQ Upper Limits
Chlorobenzilate	*10*	*190*
3,3'-Dimethylbenzidine	*10*	*190*
Butylbenzylphthalate	32	153
Bis(2-ethylhexyl)adipate	19	173
2-Acetylaminofluorene	*10*	*190*
Benzo(a)anthracene	51	123
3,3'-Dichlorobenzidine	10	214
Chysene	53	129
Bis(2-ethylhexyl)phthalate	16	190
Di-n-octylphthalate	10	200
Benzo(b)fluoranthene	44	124
7,12-Dimethylbenz(a)anthrac	*10*	*190*
Benzo(k)fluoranthene	36	147
Benzo(a)pyrene	45	136
3-Methylcholanthrene	*10*	*190*
Dibenz(a,j)acridine	*10*	*190*
Indeno(1,2,3-cd)pyrene	37	130
Dibenzo(a,h)anthracene	45	128
Benzo(g,h,i)perylene	39	134
Famphur	*10*	*190*
Kepone	*10*	*190*
a,a-Dimethylphenethylamine	*10*	*190*
p-Phenylenediamine	*10*	*190*
Hexachlorophene	*10*	*190*

Bold = Nominal limit set due to calculated limit either < 10 or > 20
Bold Italic = nominal limits set due to no statistic limits available
NS = Not Spiked
NR = Not Recovered

Method	8270	
Target List	SL LOQ Lower Limits	SL LOQ Upper Limits
Chlorobenzilate	*10*	*190*
3,3'-Dimethylbenzidine	*10*	*190*
Butylbenzylphthalate	23	158
Bis(2-ethylhexyl)adipate	10	160
2-Acetylaminofluorene	*10*	*190*
Benzo(a)anthracene	50	126
3,3'-Dichlorobenzidine	10	200
Chysene	52	121
Bis(2-ethylhexyl)phthalate	10	175
Di-n-octylphthalate	10	200
Benzo(b)fluoranthene	23	145
7,12-Dimethylbenz(a)anthrac	*10*	*190*
Benzo(k)fluoranthene	10	160
Benzo(a)pyrene	45	131
3-Methylcholanthrene	*10*	*190*
Dibenz(a,j)acridine	*10*	*190*
Indeno(1,2,3-cd)pyrene	31	152
Dibenzo(a,h)anthracene	27	159
Benzo(g,h,i)perylene	29	150
Famphur	*10*	*190*
Kepone	*10*	*190*
a,a-Dimethylphenethylamine	*10*	*190*
p-Phenylenediamine	*10*	*190*
Hexachlorophene	*10*	*190*

Bold = Nominal limit set due to calculated limit either < 10 or > 20
Bold Italic = nominal limits set due to no statistic limits available
NS = Not Spiked
NR = Not Recovered

Katahdin Analytical Services
LOQ Verification

Method:	8270-SIM		Method:	8270-SIM	
Target List	**AQ LOQ Lower Limits**	**AQ LOQ Upper Limits**	**Target List**	**SL LOQ Lower Limits**	**SL LOQ Upper Limits**
1,4-Dioxane	10	121	1,4-Dioxane	10	121
Naphthalene	33	97	Naphthalene	33	97
2-Methylnaphthalene	30	135	2-Methylnaphthalene	30	135
Acenaphthylene	39	121	Acenaphthylene	39	121
Acenaphthene	41	103	Acenaphthene	10	103
Fluorene	39	109	Fluorene	39	109
Pentachlorophenol	10	100	Pentachlorophenol	10	89
Phenanthrene	44	123	Phenanthrene	50	109
Anthracene	31	141	Anthracene	10	104
Fluoranthene	52	136	Fluoranthene	50	118
Pyrene	61	115	Pyrene	10	115
Benzo(a)anthracene	40	124	Benzo(a)anthracene	50	123
Chysene	43	138	Chysene	10	104
Benzo(b)fluoranthene	36	130	Benzo(b)fluoranthene	36	130
Benzo(k)fluoranthene	20	127	Benzo(k)fluoranthene	10	115
Benzo(a)pyrene	51	110	Benzo(a)pyrene	10	110
Indeno(1,2,3-cd)pyrene	44	129	Indeno(1,2,3-cd)pyrene	44	129
Dibenzo(a,h)anthracene	52	122	Dibenzo(a,h)anthracene	44	133
Benzo(g,h,i)perylene	48	120	Benzo(g,h,i)perylene	48	120